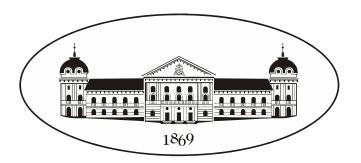


# Topic № 1 FIBRES AND YARNS; CHEMICAL TECHNOLOGIES; NANOTECHNOLOGIES











### **TEXTILE MATERIALS IN TRANSDERMAL THERAPY**

### **Daniela ATANASOVA<sup>1</sup>**, Desislava STANEVA<sup>1</sup>, Ivo GRABCHEV<sup>2</sup>

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Achievements in drug delivery systems give new impulsion to transdermal therapy used from ancient times. It can affect a number of nutritional deficiencies, leading to problems in the immune, hormonal and nervous systems, to protect cells from oxidative destruction, to influence cancer formation and therapy in diabetic patients.

Textile materials play an important role in transdermal therapy as they are a suitable matrix for different active substances and can promote their gradual release. Textile materials are characterized by a variety of composition and structure, which allows meeting the requirements for specific treatment. The easy application of biologically active textiles leads to greater comfort and less pain for the patients, reduced hospital stay and so on. Aromatherapy, antimicrobial substances and painkillers, hormone therapy, psoriasis treatment, atopic dermatitis, melanoma, etc. are some of the areas where textiles can be used as carriers.

The possible application methods of biologically active substances to textile materials are varied. The oldest one is a pad-dry-cure method. Another widespread method is by microencapsulation. In the recent years, research is aimed at new structure as cyclodextrins, aza-crown ethers, fullerenes, liposomes and polymeric micelles. Numerous functional groups and the ability to encapsulate different substances determine dendrons and dendrimer molecules as promising carriers for drug delivery. Hydrogels are also high molecular hydrophilic structures with which the textile material can be modified. They absorb a large amount of water or biological fluids and can support the delivery of medicines.

These characteristics correspond to one of the current trends in the development of materials used in transferral therapy. This is intelligence, i.e. controlled concentration and time delivery of the active substance and simultaneous visualization of the process, which can only be achieved with appropriate and purposeful modification of the textile material.

Keywords: transdermal therapy, smart textile, drug delivery

#### Acknowledgements:

The authors acknowledge Grant KOST № 1/03-2017, Fund "Scientific Research", Ministry of Education and Science of Bulgaria and Grant № 11777/2018, Scientific Research Sector of University of Chemical Technology and Metallurgy.



# COLLAGEN AND KERATIN – WASTE PRODUCTS FROM LEATHER AND TEXTILE PRODUCTION AND METHODS FOR THEIR UTILIZATION

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The production of leather and leather goods is the world's largest industrial sector, which uses byproducts. It processes the potential meat waste into high quality consumer goods. But at the same time, the leather industry is one of the most intense pollutants of the environment, releasing: carrion (decomposing subcutaneous tissue), leather, hair, soluble proteins, fats, chemicals. The textile industry also generates a significant amount of hair waste during the production and processing of wool. The leather and hair waste contains valuable raw materials: collagen and keratin.

Collagen waste includes meat waste (~ 60%). Every year, biomass over 50 billion t collagen is accumulated in the world, and only ~ 4 million t are used. Also, keratin waste exists in abundance, i.e. these are waste from the leather and textile industries, from slaughterhouses, poultry farms, etc., estimated at 5 million t globally.

Collagen and keratin isolated from leather and textile waste are applied in the following areas: 1) obtaining new materials for the leather industry; 2) in food processing and other industries (paper, furniture); 3) in agriculture as organic fertilizer components and feed mixtures; 4) in cosmetics, pharmacy; 5) in medicine such as collagen and keratin biomaterials and as biocomposites - in combination with other materials (other natural or synthetic polymers, ceramics).

We have synthesized biocomposites based on collagen and polyurethane and analyzed their biocompatibility and physico-mechanical properties, proving their application in tissue engineering.

The main methods for processing of collagen- or keratin-containing waste include various types of hydrolysis: alkaline, acidic and enzymatic. Recent studies have focused primarily on the environmental impact of these processes and the enzymatic processing of collagen containing raw materials is the most environmentally friendly.

The extraction of keratin hydrolysates is accompanied by a number of difficulties due to the nonreactivity and resistance of keratin and consequently the hydrolysis processes are carried out at drastic conditions and with the participation of aggressive reagents. In addition to peptide bonds, the presence of disulfide bonds greatly obstructs the course of these processes. It has also been proven that the pre-alkali treatment of raw materials facilitates these processes. Nevertheless there is a progress in the extraction of keratin products and their use as biomaterials. Experimental we also have obtained keratin hydrolyzates from goat hair and sheep wool using various reducing agents and various reaction conditions.

Optimizing hydrolysis procedure is of great importance to environmental protection and to receive energy-saving technologies in this field.

Keywords: collagen, keratin, hydrolizates, biocomposites



# DETERMINATION OF THE GEOMETRIC CHARACTERISTICS OF THREADS AND FABRICS THROUGH MICROSCOPE MEASURING

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#### Introduction

With the invasion of the computer technologies, the software products for capturing objects and processing their video images received a widespread application. The method for investigation of textile structures is not standardized, but is increasingly used because of its non-destructive character and possibility to measure geometric characteristics which can't be determined otherwise or which determination by conventional testing methods causes difficulties. The measurement of the characteristics in woven state (non-destructive) inevitably shows differences from the free-state (destructive) measurement, mainly due to the deformation of the threads in the weave formation.

The aim of the work is to determine the degree of deviation between the results obtained by the standardized destructive methods and the non-destructive microscopy.

#### **Experimental part**

The experiment was carried out using the following four groups of fabrics made from: 100% Cotton; 50/50% Cotton/Polyester; chemical fibers (100% PES and 100% Rayon) and 100% Wool. For each group, three representatives were selected.

The digital images were obtained and measured by means of system of Motic (Germany) including a stereomicroscope, a digital camera, and software for capturing and measuring geometric dimensions.

The diameters of the warp and weft threads, as well as the distances between them, were measured. From the distances, the threads' densities were calculated. By the gravimetric method the counts of the threads are determined and the diameters were calculated empirically.

The relative deviations between the results obtained by the standardized methods and by microscopy were determined. Correlation and regression statistical analyzes have been carried out.

#### Results

It was found that the diameters obtained by microscopic measurements are 11% higher than these received after recalculation of the count determined according to the gravimetric method. A strong statistical relation between the diameters determined by both methods (R = 0.97) was found, and therefore a linear regression equation was derived.

Regarding the thread densities in the weave, the deviations between both methods are considerably smaller: 1.5% for the warp threads' density and 4% for the weft threads' density. Due to the strong statistical dependence (R = 0.95), a regression equation was produced.

#### Discussion

The deviations between methods studied are lower for the warp thread diameters than for the weft ones, because of the higher torsion intensity and greater compactness of the warp yarns. Lower are the differences for the warp thread densities, as well. The reason is the greater uniformity of threads' warping compared to the weft insertion.

#### Conclusion

The differences between non-destructive and destructive methods for measuring of threads' and fabric geometry are determined and analysed. Linear regression equations have been derived that allow a recalculation of results obtained.

Keywords: Non-destructive analysis, microscopy, diameter of threads, threads' densities in the weave.



# INVESTIGATION THE POSSIBILITY OF CHROME FREE "WHITE" TANNING WITH DMT – II PRODUCT OF ENZYMATICALLY TREATED SHEEPSKINS

### Donika KATZAROVA<sup>1</sup>, Margarita KOLEVA<sup>1</sup>, Staneva DESISLAVA<sup>1</sup>, Ivanova DIMITRINA<sup>1</sup>

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#### Introduction

Chrome tanning has for a long time enjoyed a unique position amongst tanners all over the world and almost 90 % of all leather produced is chrome tanned.

A number of studies have been published recently suggesting that Cr(III) itself may be toxic at higher levels under certain ligand environments and also that Cr(VI) is a known carcinogen. In view of this tanners have to consider how best to modify their tanning agents and technology. For this reason, the market has shown a growing demand for "ecological" products, especially using alternative tanning agents different from chromium, such as metallic substitutes - Al, Zr, Ti, Li. Part of these guidelines is the so-called "white tanning". The aims of this work are to study the possibility of "white" tanning of enzymatically treated sheep skins with a new product DMT - II.

#### **Experimental part**

Materials, procedures and analytical methods

DMT-II - previously prepared mixed tanning agent, complex of Zr-Al-Ti; Cr tanning product; sheep skins;

Shrinkage temperature (Ts), antibacterial activity, common water removable, true pore volume and physical properties of the leathers were tested by the standard methods.

A new Zr-Al-Ti combination tanning salt (in an appropriate ligand environment) has been prepared. Leathers processed using these tanning salt exhibit shrinkage temperature within the range of 110°C to 121°C. It has been observed that the addition of Zr and Ti leads to improvement in the antimycolitic properties. In this study we compare two samples of sheep skins by changing the composition agents. The tensile strength is highest when we used Cr tanning agent.

#### Conclusions

1. The tanned leathers are suitable for production of leathers for clothing and gloves according to BDS;

2. The tanned leathers with DMT-II are suitable for the article "white" leather;

3. DMT-II product is a very suitable substitute for chromium tanning;

4. In all tanning variants, the leathers have antibacterial activity, strongly expressed in the DMT-II products;

Keywords: chrome - free tanning, sheep skins, zirconium, titanium, aluminium

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# A STUDY ON ELECTRICAL RESISTANCE OF CONDUCTIVE THREADS WITH APPLICATIONS FOR SMART TEXTILES

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#### Abstract

Nowadays the development of smart devices is a worldwide trend. In the field of the design and construction of textile products, smart devices are those with embedded electronic components with primary application in the field of medicine. Their main purpose is a real-time measurement of various parameters of the human body and the environment to evaluate both the human health and comfort. The variables that can be monitored are, for example, the skin temperature, heart rate, amount of moisture, pH, and nitrate levels among others.

The textiles and clothing play a role of a carrier phase and are preferred over other materials for several reasons: they are an insolation barrier between the human body and the environment, not expensive, easy for production, and provide comfort and convenience.

When implementing electronic components into fabrics, several problems can arise, related with both the manufacturing and the exploitation of the smart item. One of the critical issues is the secure connection between the individual modules that build the smart system. Because of the stretching of the textile (knitted or woven) during wearing, the traditional wires should be replaced by conductive threads.

There are several ways to implement a conductive thread into a textile. It can be incorporated for example in the textile during weaving, but most frequently it is sewn into the textile structure. There are two types of conductive threads. The first one is from stainless steel filaments or fibres, which can be metal-wrapped, metal-filled and metal-twisted. The second type is the traditional non-conductive filaments plated with silver or copper.

The primary parameter of the conductor is the electrical resistance, which depends on the type of the material. During wearing of the smart textile or smart clothing, it is a subject of stretching, which means that the conductor is under stress conditions.

The main goal of the present paper is to measure the electrical resistance of two type of conductive thread under various loads, reaching the limit of ultimate strength. This will give valuable information in the design stage of the smart textile system for the calculation of the designed electronic circuit, as well as the energy consumption. The conditions during the maintenance of the smart textile are also tested, such as washing and drying cycles. An analysis is made of these effects on the electrical conductivity of the threads, and especially the naturally occurred wearing process that may cause interruption of the electrical connection, particularly of coated conductive threads.

Keywords: smart textile, conductive threads, electrical resistance.

# COMPARATIVE DESCRIPTION OF THE SURFACE AND STRUCTURAL EFFECTS ON FIBRE-FORMING POLYMERS AT THE PHYSICO-CHEMICAL TREATMENT OF THE TEXTILE MATERIALS

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The physicochemical treatment (FCT) of fibrous materials accompanies the mechanical technology of textile products from fibre to fabric.

In some cases, FCT acts predominantly on the surface of the fibres, i.e. on the peripheral molecules of the textile substrate. This is the case for dyeing where chemical bonds between dye molecules and peripheral fibre molecules are obtained by ion exchange. One of the peculiar and up-to-date dyeing processes is the transfer printing. By its nature, this dyeing process adhere the microparticles of the dispersion dye onto the surface of the fibres. There is no chemical interaction between the textile substrate

and the dye substance. The adhesion between the dye and the textile is due to the short-term melting of the textile fibre peripheral layer and the penetration there of the dispersed microparticles of the dye. In order to achieve this superficial and short-lasting melt, it is necessary to heat the entire mass of the textile substrate as well as the entire fibre volume. As a result, even for surface treatment at a depth of no more than 150-200 nm, and without chemical interaction, heat treatment of the fibre-forming polymer occurs.

In other cases, FCT acts predominantly on the structure of fibreforming polymers. These are all processes of the final treatment, finishing of the raw woven fabrics. Washing, fixing and drying of fabrics have the greatest impact on their geometric dimensions. This reflects directly the sharp change in the amplitude and the denseness of the undulating deformations of the warp and weft threads. All subsequent finishing processes in various forms represent moisture heat treatment, which aims and achieves uniformity of the deformations on the fibres and the threads. Thus, the fabrics obtain a smooth and even surface.



Subject of this article is the equilibrium between the surface and structural effects of FCT on the properties and the behaviour of the fibrous materials. Aim of the work is to determine the exact parameters of the effects such as depth of penetration and degree of change of initial parameters of fibre-forming polymers. The tasks of the initial stage of studying this issue include literary research, technological observation and technology gathering.

#### Acknowledgments

The authors thank E. Miroglio EAD - Sliven for the support and assistance in the performance of the graduate work at the College - Sliven of the Technical University - Sofia and presentation of the results of the National Textile Conference in 2018.



# DEPOSITION OF SILVER COATINGS ON TEXTILE FIBRE SUBSTRATES BY MEANS OF ION-BEAM SPUTTERING IN A VACUUM MEDIUM

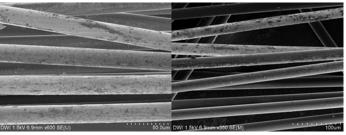
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The experiments carried out show that the deposition of metallic coatings in particular silver on textile substrates in a vacuum environment by means of an ionic plasma beam is possible and feasible. The main task, namely the deposition of thin-film silver coatings on textile fibres in the vacuum environment, was successfully achieved.

The preliminary experiments carried out in the dissertation show that the deposition of electrically conductive materials, pure silver 99, 99% on textile panels, yarns and tapes is possible, but the established electrical conductivity is insufficient to obtain low electrical resistance.

The results of the metallised fibres tests showed the preservation of the qualitative characteristics of the substrates after the ion metal metallisation, namely their flexibility, plasticity and softness typical of the textile fibres.



The plasma finishing in the vacuum medium then not only complements the final cleaning of

the fibres, but also contributes to the activation of the surface of the fibre mass. The extraction voltage should not exceed 100 V, and the plasma etching time should not be longer than 3 min.

The thickness of the metallic coating ensures the maintenance of the technical and mechanical characteristics of the fibres. The morphological and chemical analyses of the deposited coatings confirm the results obtained and facilitate the establishment of specific and strictly validated process parameters to achieve permanent metallisation and good adhesion performance.

Clear and accurate vacuum deposition parameters have been developed that can be repeatedly reproduced. Ionic plasma deposition under vacuum was the correct method for metallisation of substrates that cannot be subjected to high temperature loads by another vacuum plasma method. Because of the low process deposition temperature, this metallization method is particularly distinguished from the other methods known and described in this dissertation thesis. The experimental results presented in this dissertation thesis can be fundamental for further research on metallised textile fibres and their derivatives.

#### Acknowledgments

The authors thank E. Miroglio EAD - Sliven for the support and assistance in the performance of this doctoral work at the College - Sliven of the Technical University - Sofia and presentation of the results of the National Textile Conference in 2018.

Keywords: polyester fibres, sliver coating, ion-beam sputtering, vacuum.

# SINGLE AND BUNDLE COTTON FIBRES MECHANICAL PROPERTIES MODELLING USING ANALOGICAL MODELS

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**Abstract:** Among the many properties of cotton fibers, mechanical ones are the most important indicators to select the proper fibers for specified textile end use applications. Either the single cotton fibers properties or the spun yarn ones are related not only to their tensile properties, but also to the time dependent ones such as the creep and the stress relaxation. In addition, the frictional behavior of cotton fibers greatly influences their processing, their performance and the performance of the final product.

In this paper, two methods of cotton fibers testing are presented: single fibers and bundles. Three different types of cotton fibers were studied, having different physical properties (maturity, fineness, micronaire, length, tenacity etc.). We show that the creep behavior of cotton fibers can be assimilated to a Voigt model in series with a spring and that the difference in the behavior between the single fibers and bundles is related to the inter-fiber friction.

Studying the behavior and the relationships between single and bundle cotton fibers mechanical properties is very crucial. In fact, single fibers are the fundamental units of a span yarn [1]. Any study of a yarn model must include the parameters of the fibers and their relationships. In general, fibers physical properties (fineness, diameter, shape factor and length) contribute to yarn strength through two factors: fiber strength and inter-fiber friction. For this purpose, we



aim to analogically model the cotton fibers relationships and to study the inter-fiber friction in the bundle of cotton fibers.

Cotton fibers contain natural polymers (90% of cellulose), and therefore they exhibit a viscoelastic behavior [2]. This mechanical behavior can be adjusted using analogical models consisting of elements such as Hook springs, Newton dashpots, as shown in table 1, which could simulate the mechanical behavior of the material under mechanical stress when correctly combined.

These models are very useful to clarify how fiber behaves. They can be assembled both in series or in parallel or in mixed groups [3]. Thus, more complex mechanical responses can be simulated to illustrate the behavior of the material submitted to static test (tensile test) or a time dependent one (creep or stress relaxation tests).

Keywords: Bundles, cotton, inter-fiber friction, modelling, single fibers.



# CHROME FREE TANNING - AN INVESTIGATION OF ALUMINIUM COMBINATION TANNING WITH SUMAC, MIXED SUMAC-METAL AND CLAY PRODUCTS

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#### Introduction

Currently, there is a globally growing requirement for wet-white leather products, including automotive leathers, upholstery leather and garment leather with considerable high-quality and high value-added characters. The wet white tanning approach produces non-polluting and non-toxic solid wastes and has substantial environmental benefits because of its significant reductions in hazardous waste disposal and the level of chemicals in the effluents. Therefore, wet-white tanning is considered as a feasible alternative to conventional chrome tanning for chrome free leather manufacture.

#### **Experimental part**

Materials, procedures and analytical methods

C0 - previously prepared vegetable (sumac) tanning agent

C24 - previously prepared mixed vegetable-metal tanning agent, complex of TiO2 with sumac extract Al tanning agents- Lutan B, Tanit AGS, clay product, sheep skins

In this study we compare three samples of sheep skins tanned in three variants by changing the composition agents. Shrinkage temperature (Ts), antimicrobial activity, anti- UV property and absorbtion for colometric measuments, common water removable, true pore volume and physical properties of the leathers were tested by the standart methods.

#### **Results and discussion**

Tanning complexes in new products have a low degree of cross-linking of collagen of the dermis. Shrinkage temperature (Ts) of the tanned three sheep skins is within the range of  $60^{\circ}$ C to  $64^{\circ}$ C. The tensile strength are highest when we used the Al tanning agent in the pickle bath. The results obtained convincingly prove the antimycotic properties of the products and their anti-UV property of the proposed treatment.

#### Conclusions

1. The tested tanning agents mostly are deposited on the collagen fibrers

2. The tanned leathers with Al tanning agent in the pickle bath leads to their lightening

3. The wet -white leather exibits higher absorbance or lower trasmittance in the near UV region and exhibits the enhaced anti-UV property

4. The titanium content of the complex C24 gives an increased antibacterial effect

**Keywords:** chrome - free tanning, sheep skins, complexes, vegetable tannin, titanium, aluminium; clay

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# CURCUMIN-LOADED FIBROUS MEMBRANES WITH COMPLEX ARCHITECTURE AND BIOLOGICAL ACTIVITIES

### Mariya SPASOVA<sup>1</sup>, Petya TSEKOVA<sup>1</sup>, Nevena MANOLOVA<sup>1</sup>, Iliya RASHKOV<sup>1</sup>, Nadya MARKOVA<sup>2</sup>, Ani GEORGIEVA<sup>3</sup> and Reneta TOSHKOVA<sup>3</sup>

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**Introduction** Curcumin (Curc) is a naturally occurring polyphenolic compound with antiinflammatory, antibacterial and antitumor activities. However, its clinical application is limited by its poor bioavailability related to its extremely low water solubility. In the recent years, electrospinning has emerged as a very suitable technique for preparation of drug-loaded polymeric materials. It has been shown that electrospun fibrous materials are suitable carriers for enhancing the bioavailability of Curc [1, 2].

**Experimental part** Cellulose acetate (CA, Aldrich) with  $\overline{Mn} = 30\ 000\ g/mol$  and DS 39.8%, polyvinylpyrrolidone (PVP, Fluka) with Mr 24 000 g/mol and curcumin (Merck) were used. Acetone (Sigma-Aldrich) and ethanol (Sigma-Aldrich) of analytical grade of purity were used.

**Results** Curc release profile was modulated by varying the electrospinning/electrospraying techniques and the polymer matrix composition. The incorporation of PVP facilitated Curc release. The enhanced Curc solubility is related to its ability to form hydrogen bonds with certain polymers such as: PVP, polyethylene glycol and CA, undergoing transition from crystalline to amorphous state.

**Discussion** Our findings reveal that the use of electrospinning in conjunction with electrospraying provides more possibility to modulate the composition and architecture of the obtained materials. It was found that the incorporation of Curc imparted antibacterial properties against the pathogenic microorganisms S. aureus and E. coli and antitumor properties to the obtained novel membranes.

**Conclusion** Curc-loaded membranes have been successfully prepared by conjunction of electrospinning and electrospraying techniques. The incorporation of the water-soluble polymer modulates the wetting of the membranes and facilitates the dissolution and release of Curc. The obtained fibrous membranes exhibit antibacterial and antitumor activities.

Keywords: curcumin, fibrous membranes, antibacterial and antitumor activities.

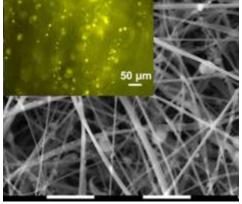
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# POTENTIAL OF THE DENDRIMER ARCHITECTURE AS A NOVEL ANTIMICROBIAL AGENT FOR TEXTILE

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In the recent years, a new class of antimicrobial agents has been actively studied. Unlike low and high molecular compounds, the dendrimers have a higher antimicrobial activity at low concentration. The large numbers of functional groups on the dendrimer surface allow the binding of many small molecules with antimicrobial activity. Thus, a large dose of a biologically active substance can be delivered with only one molecule. The antimicrobial effect may additionally be enhanced by reaction of the internal functional groups with metal ions or by encapsulating nanoparticles or other biological active substances. This allows better control over the processes of bacterial resistance and biofilm formation.

The development of technologies for the application of the dendrimers in the production of antimicrobial textile materials is a new current direction with a large multi-billion market. The challenge is to achieve an appropriate biocide activity combined with stability under conditions of use and maintenance while preserving the specific properties of the textile.

In the production of antimicrobial textile material, a number of factors should be considered, such as the application areas of the material, the type of textile material, the chemical structure of dendrimer and its concentration, the methods for the application of the dendrimer to the textile material, the preparation of the metal complexes and the reduction of metal ions to nanoparticles.

Keywords: textile, antimicrobial, dendrimer, metallodendrimer, nanoparticles

#### Acknowledgements:

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### CAFFEIC ACID PHENETHYL ESTER- CONTAINING ELECTROSPUN FIBROUS MATERIALS WITH ANTIBACTERIAL AND ANTIOXIDANT PROPERTIES

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**Introduction** During the last decade, the use of electrospinning for the fabrication of drug-loaded nanofibrous materials for biomedical applications such as wound healing dressings has evoked considerable interest [1]. The size-related properties of these materials, and the possibility to control their properties by controlling their architecture, the fibers morphology, porosity and composition, can lead to control on the drug release, enhancement of the drug bioavailability and avoiding side effects. Electrospun fibrous materials are very promising for topical delivery of drugs. Caffeic acid phenethyl ester (CAPE) is one of the main components in honeybee propolis. While possessing a number of valuable biological properties such as antioxidant and antibacterial activities, it is poorly soluble in water and body fluids, which predetermines its low bioavailability. Therefore finding routes to preparation of CAPE-loaded fibrous materials is of particular interest.

**Experimental part** CAPE-containing fibrous materials were prepared according to the procedure described earlier [2]. The morphology of the fibrous materials was evaluated by scanning electron microscopy (SEM) with Jeol JSM-5510 (Jeol Ltd., Japan). The in vitro dissolution test was carried out spectrophotometrically. The antibacterial activity of the CAPE-containing fibrous materials against S. aureus and E. coli bacteria was evaluated by using the viable cell-counting method. The antioxidant activity of CAPE-containing mats was measured using the DPPH assay.

**Results and Discussion** In the present study novel fibrous materials with enhanced aqueous solubility of CAPE and with facilitated CAPE release were successfully prepared. These materials were obtained from poly(3-hydroxybutyrate) (PHB) by applying electrospinning or electrospinning combined with dip-coating. The release of CAPE was affected by the composition of the polymer matrix and the method of preparation. Incorporation of polyvinylpyrrolidone (PVP) in the bulk of the fibers or in a coating on the fiber surface, and exploitation of the ability of this polymer to form hydrogen bonds with CAPE, resulted in enhancement of CAPE solubility. It was found that CAPE when incorporated in the bulk of the fibers or in a PVP coating on the fiber surface was in the amorphous state which is favorable for utilization in drug dosage forms. Microbiological tests revealed that the fibrous materials containing CAPE exhibited biocidal effect against the pathogenic S. aureus and E. coli bacteria. The obtained materials exhibited good antioxidant activity similar to that of free CAPE.

**Conclusion** The results suggest that the obtained novel fibrous materials containing CAPE are potential candidates for wound dressing applications.

Keywords: electrospinning, caffeic acid phenethyl ester, antibacterial fibers, antioxidant fibers

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### NOVEL ELECTROSPUN FIBROUS MATERIALS OF ALIPHATIC POLYESTERS AND THEIR STERILIZATION - CHALLENGES AND SOLUTIONS

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**Introduction** Aliphatic polyesters have become one of the most preferred polymers for developing new generation devices for biomedical applications. Electrospinning has justifiably found its place among the most effective techniques for designing diverse micro- and nanofibrous materials. Electrospinning renders possible obtaining fibrous materials with no significant phase separation from otherwise immiscible poly(L-lactide)(PLLA) and poly(butylene succinate) (PBS) [1]. Since electrospun fibrous materials from such polyesters are very convenient for use in biomedicine, their sterilization is often necessary. Sterilization of polymeric materials commonly meets an obstacle related to their thermal and hydrolytic stability. The aim of the present work is to propose a suitable approach to overcome this problem.

**Experimental part** For the synthesis of PDLA-b-PBS copolymers poly(D-lactide) (PDLA) and PBS with Mn 4000 and 2000 g/mol, respectively, were used at a PDLA/PBS molar ratio = 9/1 or 1/1. The copolymers and high-molar-mass poly(L-lactide) (HPLLA, Unitika 6201, MW=95000 g/mol) were used for preparation of stereocomplex-based fibrous scaffolds [2].

**Results** By electrospinning new fibrous materials from stereocomplex between HPLLA and PDLAb-PBS were prepared. The stereocomplex formation was evaluated by DSC and X-ray analyses. The thermal properties and the crystal structure of the new stereocomplex-based fibrous materials depended on PBS content in the copolymers. The thermal degradation profile of the mats strongly depended on PBS content. Two methods for thermal sterilization were applied under standard conditions - autoclaving at 121°C for 20 min at 1 atm, and dry heating at 160°C for 2 hours. Microbiological tests revealed that the fibrous mats were sterile.

**Discussion** The melting temperature of stereocomplexes is significantly higher than that of the enantiomeric polylactides and exceeds 200°C. Therefore, it was appropriate to exploit this advantageous property and to prepare electrospun materials suitable for sterilization. It was proved that thermal treatment was not detrimental to the morphology of the fibrous materials.

**Conclusion** The proposed use of stereocomplex formation between aliphatic (co)polyesters for the preparation of fibrous materials by electrospinning turns a promising route to the fabrication of sterilizable fibrous scaffolds for use in biomedicine.

Keywords: electrospinning, stereocomplex, aliphatic polyesters, sterilization

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### FROM MICROBEADS TO MICROFIBERS BY ELECTROSPRAYING/ELECTROSPINNING – EFFECT OF CELLULOSE ACETATE CONCENTRATION

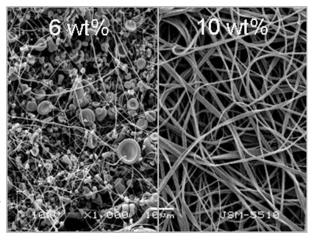
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**Introduction** Cellulose is the most abundant naturally occurring polysaccharide. Cellulose can be converted upon acetylation to cellulose acetate. The advantages of the resultant cellulose acetate is that it could be easily dissolved or melt and could be shaped into fibers, films, sheets, tubes, pellets or other products. In recent decades, great attention has been paid to fibers from cellulose and cellulose derivatives due to their low cost, lightweight, easy processing, biodegradability, good mechanical and barrier properties and recycling. Recently, the electrospinning technique has been considered as a versatile and effective method for manufacturing nanofibrous materials with high surface-to-volume ratio and multi-porous structures.

**Experimental part** Cellulose acetate (CA, Aldrich) with  $\overline{Mn} = 30\ 000\ \text{g/mol}$  and DS 39.8%, was used. Acetone was of analytical grade of purity and was purchased from Sigma-Aldrich.

**Results** Fibrous materials were prepared from CA solutions by conjunction of electrospraying with electrospinning. The polymer concentration was varied: 6, 8, 10 and 17 wt%. The effect of the polymer concentration on the viscosity of the spinning solutions and on the morphology of the obtained electrospun mats was evaluated. Scanning electron microscopy (SEM) images revealed that beaded fibrous structure was observed at concentrations below 10 wt%. The experimental results showed that defect-free fibers with mean fiber diameter of  $780 \pm 110$  nm were obtained at 10 wt% concentration of CA in acetone/water. Electrospinning of CA solution with concentration of 17 wt% resulted in preparation of fibers with large diameters (~3,5 µm).



**Discussion** Our findings reveal that the viscosity of the spinning solutions and the morphology of the obtained fibrous mats were greatly influenced by the CA concentration in the solutions. The formation of beads at lower polymer concentrations and the increase in fiber diameter with increase in solution concentration was attributed to the changes in viscosity of the solution. Solution viscosity is related to the extent of polymer chain molecules entanglement within a solution.

**Conclusion** CA microbeads and fibers were prepared by electrospraying/electrospinning. The solution concentration was varied. The beads size and fiber diameter were greatly influenced by the concentration and the viscosity of the solution. The optimal conditions for preparation of defect-free CA fibers were found: polymer concentration of 10 wt% (dynamic viscosity - 122 cP), tip-to-collector distance - 15 cm and applied voltage of 25 kV.

Keywords: cellulose acetate, concentration, electrospraying, electrospinning, morphology.



### STUDY THE IMPACT OF NANOPOWDERS Si<sub>2</sub>C ON THE BALLISTIC CHARACTERISTICS OF ULTRA-HIGH-MOLECULAR-WEIGHT POLYETHYLENE

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**Introduction** Ballistic protection products, it is necessary to use materials to create a lightweight armor that is light and provides comfort when wearing it. Combining the properties of metal alloys, fibers, polymer materials, textiles, nanopowders by reinforcing them with a high polymer matrix can provide high ballistic protection.

The aim of this study is to create a lightweight model of product for individual ballistic protection by combining the properties of heterogeneous materials into a single composite system that provides reliable protection and wearing comfort. High molecular weight polyethylene, nanopowders  $Si_2C$ , polymers with a high strength are embedded in a single system without significantly changing parameters such as the weight, thickness and density.

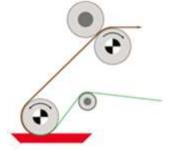
**Materials and experimental procedure** Created is a composite system of Polyvinyl butyral (PVB), micro particles (Si<sub>2</sub>C) applied to high molecular weight polyethylene (UHMWPE).

The ultra-high molecular polyethylene (UHMWPE) is a multilayer fabric synthetic produced by Dyneema® SD. The fabric is 1 mm thick and has a mass of about 140 g/m2. The construction of multilayer polyethylene fabric is a reinforcement that gives mechanical strength and elasticity to the individual ballistic protection means.

Polyvinyl butyral (BUTVAR B-98), manufactured by ACROS Organics (The Netherlands), is a white powder dissolved in ethanol to create a highly viscose resin which after curing creates a strengthening matrix.

Silicon carbide (Si<sub>2</sub>C) have particle size Dpart =  $0 \div 5 \mu$  (microns). is a material that gives high strength and chemical stability.

Technological possibilities that eligible materials for applying the resin, such as PVB, with  $Si_2C$ , which improve adhesion on Dyneema® SD fibers is showed on *Figure 1*.



*Figure 1* Deeping the textile in matrix solution

**Keywords:** Armor, ballistic protection,  $Si_2C$ , polyvinylbuteral, ultra-high molecular polyethylene (UHMWPE).

### INITIAL STUDY OF THE MORPHOLOGICAL AND GEOMETRIC PARAMETERS OF KAPOK FIBRES

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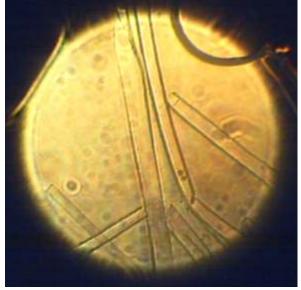
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Textile products of cotton and cotton-type fibrous blends have a wide variety of fibre combinations, construction and colour. One of the current trends for their diversification when preserving the natural origin of the raw materials is the incorporation of exotic and atypical plant fibres. Such a type of fibre is

the kapok. Its filamentous plant Ceiba pentandra grows to 73 m, and the commercial tree is most heavily cultivated in the rainforests of Asia, notably in Java, the Philippines, Malaysia, and Hainan Island in China, as well as in South America. The tree grows in the tropical regions in the countries: Myanmar, India, China, Bangladesh and others.

Previous attempts to apply the kapok fibres to cotton spinning have identified the following main dependencies: Applying more than 20% of the kapok fibres abruptly reduces the technology patency of the spinning blend. Kapok fibres are light, very buoyant, resilient, resistant to water, but it is very flammable, so they are hard to process in the spinning process stream, and usually make healthier working conditions worse.

Regardless of the limitations mentioned above, the kapok fibres add extra softness and glow to the yarn. Textile products gain a noble appearance and increased commercial value.



The major problem of the industrial application of Kapok fibres lies in the inadequate knowledge of their properties and technical characteristics. There is an initiative to study these fibres, which in its initial stage focuses on a description of their morphology and thickness. Microscopic photographs show that these fibres are fine and have a smooth almost cylindrical surface. Laboratory measurements of their finesse with FADA showed an average thickness of 10.23  $\mu$ m, with a relatively large variation coefficient of 35.0%.

Subject of this article are the morphology description and the geometric parameters of the kapok fibres. The aim of the work is to complementary study the properties and characteristics of these fibres as well as their possible application in a card spinning mill with conventional technological equipment. Implementation tasks include a literary study, a laboratory test of available experimental samples, and a metrological description of the fibres.

**Acknowledgments** The authors thank E. Miroglio EAD - Sliven for the support and assistance in the performance of this post-doctoral work at the College - Sliven of the Technical University - Sofia and presentation of the results of the National Textile Conference in 2018.

Keywords: atypical fibrous rows, kapok fibres, carded spinning.



# NEW TRENDS IN PRODUCTION OF NONFLAMMABLE TEXTILE MATERIALS

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The development of new technologies for the production of non-flammable textile materials is more current nowadays than ever before. Despite the achievements in this area and the availability of a large number of excellent quality products, a rethinking of their applicability from the point of view of health, ecology and recycling is under way.

The task is relatively difficult because the new substances, the technology for their application to textile materials and their consumer qualities must meet a number of requirements.

Halogen or formaldehyde containing or realizing substances have a restriction on use. The new trends in flame retardants are related to the achievements of nanotechnology and the use of natural phosphorus containing polymers (phytic acid, casein, DNA, etc.). Different methods are used for their application as pad-dry-cure method, knife over roll method, layer-by-layer assembly, sol-gel process, covalent attachment, plasma deposition.

The other trend in functionalization of textile materials is the achievement of multifunctionality. There are already many examples of obtained simultaneous flammability and improved other custom properties as colour, antibacterial properties, self-cleaning ability, etc.

Keywords: non-flammable textile, flame retardants, polymer nanocomposite

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# ELECTROSPINNING USING AN ORIGINAL "4 IN 1" COLLECTOR

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**Introduction** The conventional electrospinning set-up by using static collector enables the preparation of only randomly oriented and isotropic structures in the form of a non-woven textile because of the chaotic and whipping movement of the electrospinning jet. To overcome this limitation of the electrospinning set-up, different devices and modifications of the set-up that yield fibrous materials with defined and oriented structure and functionality have been developed. In most cases, their construction is aimed at modifying the jet movement by controlling the electric field distribution. Subsequently, the rotating drum collector has been additionally modified; as a result rotating "string" and "blade" drum collectors have been constructed. In these cases, highly aligned fibers in the gaps between the strings or blades have been collected. The aim of the present study is to create a rotating drum collector that combines all of these modifications in one.

**Experimental part** The novel, optimized and highly efficient "4 in 1" collector was designed, constructed and put into operation to the available in the LBAP-IP-BAS electrospinning set-up by applying non-conventional and innovative approaches.

**Results** The originally designed rotating drum collector that combines function of four types of collectors - conventional (smooth aluminium sheet), blade (steel), string (steel) and grid (steel), were successfully constructed and put into operation. The design of the collector allows easily configuration in these four types thus enabling fast and effective fabrication of micro- and nanofibers with tailored alignment and patterned structure. Furthermore, the developed collector broadened significantly the electrospinning capabilities and enabled the preparation of diverse in design non-woven textiles.

**Discussion** The special construction of the carrying rings allows the aluminium sheet to be easily replaced by blades. Moreover, this construction allows the blades to be easily replaced by strings, as well the strings to be easily replaced by grid. The new "4 in 1" rotating drum collector was successfully tested for the preparation of a series of electrospun non-woven materials from aligned poly(3-hydroxybutyrate) fibers with patterned architecture. In this way, the effect of the collector design and the fibers alignment degree on the mechanical properties of polymer non-woven textile might be easily studied and evaluated.

**Conclusion** It was shown that combining functions of four types of collectors - conventional (smooth aluminium sheet), blade (steel), string (steel), and grid (steel), an original "4 in 1" rotating drum collector was successfully designed, constructed and put into operation. The special construction that includes carrying rings allows facile replacement of aluminium sheet with blades, strings or grid. This original collector provides deeper insight into the electrospinning set-up and constitutes a good basis for transfer of the developed innovative approach for preparation of patterned textile from laboratory to industrial scale.

Keywords: electrospinning, "4 in 1" rotating drum collector, poly(3-hydroxybutyrate)

Acknowledgment: Financial support from the National Science Fund (Grant DN 09/2, 14.12.2016) is kindly acknowledged.



# FLAME RETARDANT FINISH FOR COTTON AND COTTON BLEND FABRICS

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#### Abstract

Cotton is the one of the most important textile fibre because of its comfort and worldwide consumption. However, it ignites easily and is frequently implicated in fire. Since, considerable efforts have been made to develop flame-retardant cotton. The flame-retardant cotton fabrics must be durable to washing cycles, eco-friendly and have a good physical mechanical properties. This article summarizes an overview of the factors related to the requirements of flame retardant cotton fabrics such as chemical agents and technological parameters.

This section also introduces some of the new chemicals and techniques used in the fire retardant treatment for cotton fabric. They allow flame retardant treated fabrics to have more durable fire resistance and better mechanical properties while still being environmentally friendly.

In the 2 and 3 sections of this article, the experimental results of study on flame retardant treatment for cotton and cotton polyester blend fabrics of the authors are presented. The experimental results show that flame retardant cotton and cotton polyester blend fabrics, which was received from this research have a good flame retardancy, but it has limited durability to wash, moreover, after treatment, mechanical strength of fabric was reduced. In order to have the durable flame retardant, eco-friendly cotton fabric, the study should be continued in the direction of using environmentally friendly chemicals and in treatment process, it should avoid the conditions that may adversely affect the mechanical strength of the fabric. Plasma application in flame retardant treatment of fabric may be a good solution to could meet all requirements of fabric.

**Keywords:** Cotton fabric, cotton polyester blend fabric, flame retardant agent, cross-link agent, Organophosphorus flam retardant agent.





