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# ANTIMICROBIAL FINISHING OF TEXTILES USING EMULSIONS BASED ON NATURAL PRODUCTS

**CHIRILĂ Laura[1], VASILE Georgiana[1], CONSTANTINESCU R. Rodica[2], OLARU Sabina[1], POPESCU Alina[1], STAN Miruna[3,4]**

[1] National Research & Development Institute for Textiles and Leather, 16 Lucretiu Patrascanu Street, 030508, Sector 3, Bucharest, Romania

[2] National Research & Development Institute for Textiles and Leather, Leather and Footwear Research Institute (ICPI) Division, 93 Ion Minulescu Street, 031215, Bucharest, Romania

[3] Department of Biochemistry and Molecular Biology, Faculty of Biology, University of Bucharest, 91-95 Spl. Independentei, 050095 Bucharest, Romania, e-mail: miruna.stan@bio.unibuc.ro

[4] Research Institute of the University of Bucharest–ICUB, University of Bucharest, 91–95 Splaiul Independentei, 050095 Bucharest, Romania

Corresponding author: Vasile, Georgiana, E-mail address: georgiana.vasile@incdtp.ro

***Abstract:*** *Bioactive dressings are obtained using substances which release bioactive compounds. A way of immobilization active ingredients on various textile materials is through emulsions based on natural polymers. Active ingredients, like essential oils and propolis play an important role in the inhibition and prevention of bacterial growth. Chitosan and xanthan gum are suitable for essential oil entrapment due to their biodegradability, low product costs and environmentally friendly production processes. The aim of this paper is to highlight the antimicrobial activity and biocompatibility of textile materials treated with cinnamon essential oil and propolis based emulsions. In this respect, experimental samples of oil-in-water emulsions type, based on chitosan-propolis-cinnamon essential oil and xanthan-propolis-cinnamon essential oil were prepared and then were immobilized on woven fabrics with different fiber compositions. The functionalized textile materials were characterized in terms of their physical-mechanical and physical-chemical characteristics, antibacterial activity and biocompatibility point of view. From the corroboration of the obtained data it was found that the obtained samples are more hydrophobic than the untreated material and the bioactive polymeric systems have shown antibacterial activity for both gram positive bacteria (S. aureus) and gram negative bacteria (E. coli) test strains. The in vitro biocompatibility evaluation on human skin cells confirmed the absence of cytotoxicity after the short-term exposure. Also, the treated samples displayed a good biocompatibility without skin irritations.*

***Keywords:*** *bioactive textiles, propolis, chitosan, xanthan, antibacterial activity, biocompatibility*

## 1. INTRODUCTION

Skin provides the barrier between the body and the environment and the first line of defence against different noxious agents. Atopic dermatitis is a chronic relapsing inflammatory skin disease which usually starts during the first years of life. There are many factors known to worsen the disease, including food and inhalant allergens, climatic factors, skin infections due to *Staphylococcus aureus*, stress, chemical and physical irritants. In the management of AD, the correct approach requires a combination of multiple treatments to identify and eliminate trigger factors, and to improve the alteration of the skin barrier. Dressings can be an effective barrier against persistent scratching, allowing more rapid improvement of the lesions, and may reduce the external source of skin irritants and bacterial infection. Traditional dressings, such as material gauze and bandages, are the most common products to treat wounds, different skin problems, providing a protective barrier against microorganisms.

Herbal remedies are used for the control of bacterial infections [1]. The essential oils are complex mixture of chemical compounds sensitive to oxygen, light and high temperature. They are hydrophobic and therefore insoluble in water, which reduces their bioavailability and absorption in to the body. The encapsulation of essential oils in different matrices is necessary in order to prevent this inconvenience [2].

*Cinnamomum verum* (cinnamon) essential oil is known to have antioxidant, anti-inflammatory, and antimicrobial properties [1]. Propolis is a resinous substance of bees *Apis mellifera var. carnica*. Based on pharmacology, flavonoids are the most important. They are attributed many therapeutic effects such as antioxidant, antibacterial, antiviral, anti-inflammatory and antitumor [3].

A natural polymer used successfully in the biomedical field is chitosan, derived from chitin. It has the property to form films, is easy to chemically modify and has a high mechanical strength [4]. Chitosan exhibits excellent biocompatibility, biodegradability, nontoxicity and anti-microbial activity. Also, chitosan has an adhesive nature, antifungal, bactericidal feature, and oxygen permeability. Because of these immense activities, chitosan and its composites show good positive impacts on wound healing [5]. Xanthan gum is a microbial polysaccharide that has been widely used in medicine, food technology, chemical industry, textile and other fields, due to its desirable thickening, suspension, thermal resistance, acid and alkali resistance, electrolyte resistance and other properties [6].

The main goal of this study was to develop biofuntional textile materials designed for the treatment of inflammatory skin conditions and to enable the overall finishing processes influence on the physical-mechanical and physical-chemical characteristics, antibacterial and biological properties**.**

## 2. EXPERIMENTAL PART

### 2.1. Materials

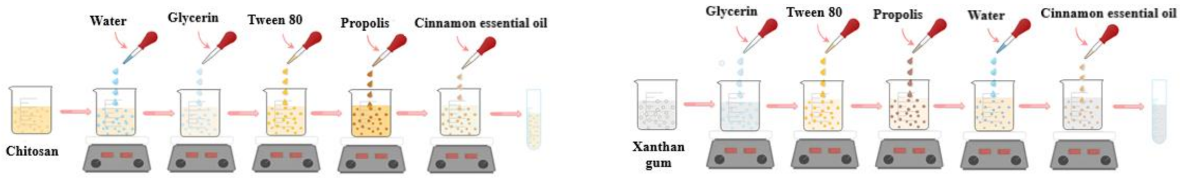
For the experiments, the following materials were used: chitosan with low molecular weight (Fluka Chemie GmbH, Switzerland) and xanthan gum (Mayam, Romania) as agents for incorporating the active principles, Tween 80 (Sigma Aldrich, Germania) as emulsifying surfactant, vegetable glycerin (SC Herbavit SRL Romania) as wetting solubilizer, cinnamon leaf essential oil (Adams Supplements, Romania) and propolis tincture of 70% ethanolic solution (Larix SA, Romania) as active ingredients, acetic acid (96%) (Consors, Romania), distilled water. Two scoured and bleached plain weave fabrics made from 100% cotton fixed at warp direction were used for the functionalization processes. The main characteristics of the fabrics are given in **Table 1**.

### 2.2. Preparation of polymeric bioactive systems

Oil-in-water emulsions were prepared by mechanical emulsification, the preparation methodology and their characterization being presented in previously our studies [7,8]. The succession of the preparation stages of polymeric bioactive systems is presented in **Fig. 1**.

### 2.3. Immobilization of bioactive polymeric systems on the textile materials

In this sense, the 2 polymeric systems were immobilized on the textile supports by the padding method in the following conditions: 3 passes, squeezing degree 85%, squeezing pressure 2.7 bars. The treated textile substrates were subsequently subjected to the drying operation at 50°C for 3 minutes. The codification of the experimental variants is presented in the **Table 1**.




a) b)

***Fig. 1:*** *The succession of the preparation stages of the bioactive systems: a. chitosan-propolis-cinnamon essential oil; b. xanthan-propolis-cinnamon essential oil*

***Table 1:*** Coding of textile materials treated with bioactive systems

| Code | Fibrous composition | | Mass [g/m$^2$] | Carrier system | Experimental variants |
|---|---|---|---|---|---|
| | Warp | Weft | | | |
| **V1** | 100% cotton, Nm50/2 | 100% acetate, 130dtex | 136 | chitosan-propolis-cinnamon essential oil | **V1C** |
| **V2** | 100% cotton, Nm50/2 | 100% lenpur, Nm 34/1 | 172 | chitosan-propolis-cinnamon essential oil | **V2C** |
| **V1** | 100% cotton, Nm50/2 | 100% acetate, 130dtex | 136 | xanthan-propolis-cinnamon essential oil | **V1X** |
| **V2** | 100% cotton, Nm50/2 | 100% lenpur, Nm 34/1 | 172 | xanthan-propolis-cinnamon essential oil | **V2X** |

### 2.4. Methods

#### 2.4.1. Testing the physical-mechanical and physical-chemical properties

The treated fabrics were characterized in terms of the main physical-chemical and physical-mechanical characteristics, respectively: mass per unit area (SR EN 12127-2003), hydrophilicity (drop test method according with SR 12751/1989 standard), water vapour permeability (STAS 9005: 1979), permeability to air (SR EN ISO 9237: 1999).

#### 2.4.2. Evaluation of antibacterial activity

For testing the antimicrobial activity, the diffusion agar method was used in according to the standard SR EN ISO 20645:2005, with cultures in 24-hour liquid medium of ATCC 11229 *Escherichia coli* (Gram-negative) and ATCC 6538 *Staphylococcus aureus* (Gram-positive) test strains.

#### 2.4.3. Biocompatibility

To evaluate biocompatibility, the MTT test (based on the reduction of a yellow tetrazolium salt- 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide or MTT, to purple formazan crystals by metabolically active cells) was performed to determine cell viability and the level of nitric oxide (NO) released in the culture medium was determined. Preliminary steps were required to perform these tests that consists in cultivation of HaCaT keratinocytes and exposure them to textile materials in 24-well plates and in cell culture inserts. All results were calculated with mean values of at least three independent experiments (n = 3). The data were representative of the non-impregnated control.

## 3. RESULTS AND DISCUSSION

### 3.1. Physical-mechanical and physical-chemical properties

The comparative analysis of the measured characteristics for the textile materials treated with both carrier systems, presented in **Table 2,** highlighted that the functionalization treatments generates an increase of the mass characteristics due to the amount of polymeric substances immobilized on the surface of the textile fabrics that remain fixed to the textile support at the end of the process, the growing being differentiated depending on the fibrous composition and to type the polymeric system used for the conferring of healing properties. Air permeability of functionalized samples registers much lower ratio compared to untreated ones, indicating a decrease in comfort properties for all experimental variants. The performed functionalization treatments lead to the decrease of the water vapour permeability, there being a small insignificant variation between the two variants of textile structures. The functionalization treatments, applied on both textile structures, performed with the polymeric system based on chitosan leads to a more accentuated decrease of the hydrophilicity compared to the treatment performed with the polymeric system based on xanthan.

***Table 2:*** Physical-mechanical and physical-chemical characteristics of the treated samples

| **Code** | **Fibrous composition** | | **Mass ($g/m^2$)** | **Air permeability ($l/m^2/sec$)** | **Water vapour permeability (%)** | **Hydrophilicity (s)** |
|---|---|---|---|---|---|---|
| | **Warp** | **Weft** | | | | |
| **V1** | 100% cotton, Nm 50/2 | 100% acetate, 130 dtex | 136 | 260.4 | 42.4 | instantaneous |
| **V1C** | | | 169 | 116.4 | 25.8 | 6.95 |
| **V1X** | | | 171 | 85.02 | 29.9 | 1.81 |
| **V2** | 100% cotton, Nm 50/2 | 100% juniper, Nm 34/1 | 172 | 386 | 45.5 | instantaneous |
| **V2C** | | | 201 | 349.2 | 27.5 | 5 |
| **V2X** | | | 199 | 261 | 30.7 | 0.98 |

### 3.2. Evaluation of antibacterial activity

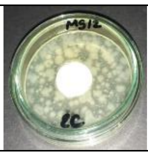
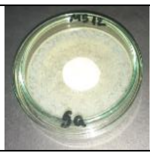
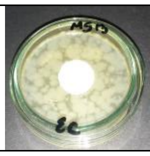
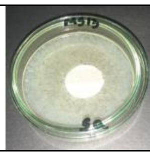
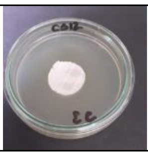
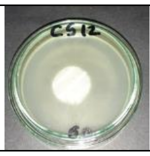
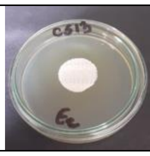
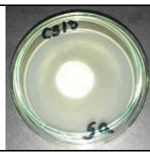
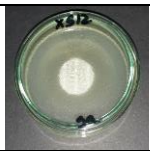
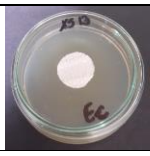
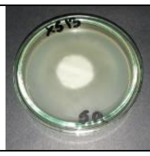
The evaluation of antibacterial activity is presented in **Table 3** and the images of the Petri dishes registered after 24 hours of incubation are shown in **Table 4**.

***Table 3:*** Evaluation of antibacterial activity

| **Code** | **Inhibition zone (mm)** | | **Evaluation** | |
|---|---|---|---|---|
| | *E. coli* | *S. aureus* | *E. coli* | *S. aureus* |
| **V1** | 0 | 0 | Insufficient effect | Insufficient effect |
| **V1C** | 16 | 11 | Satisfactory effect | Satisfactory effect |
| **V1X** | 5 | 15 | Satisfactory effect | Satisfactory effect |
| **V2** | 0 | 0 | Insufficient effect | Insufficient effect |
| **V2C** | 16 | 10 | Satisfactory effect | Satisfactory effect |
| **V2X** | 16 | 13.5 | Satisfactory effect | Satisfactory effect |

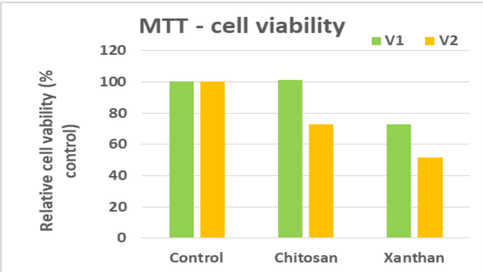
Regardless of the textile structure used to obtain biomaterials designed for the treatment of inflammatory diseases, textile structures treated with bioactive polimeric systems have an antibacterial effect against both test strains, with inhibition zones between 5 mm (V1X in the presence of *E. coli*) and 16 mm (V2X, V1C, V2C in the presence of *E. coli*). In the case of untreated textile structures, considered control, the test strains had a significant development, the inhibition zone around the textile samples being absent.

***Table 4:*** Images of Petri dishes after the exposure to the *E. coli* and *S. aureus* strains of the V1, V2 woven fabrics before and after the functionalization

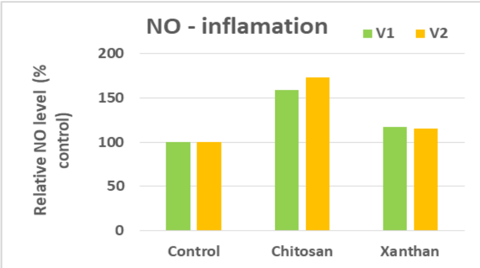
| ***E coli*** | ***S aureus*** | ***E coli*** | ***S aureus*** |
|---|---|---|---|
| **V1** | | **V2** | |
|  |  |  |  |
| **V1C** | | **V2C** | |
|  |  |  |  |
| **V1X** | | **V2X** | |
|  |  |  |  |

### 3.3. Biocompatibility

Based on the biocompatibility tests (**Fig. 2.a**), it was revealed that in the presence of functionalized textile materials, the fibroblasts cultured on inserts maintained their viability. In the case of keratinocytes cultured in the presence of textiles materials treated with chitosan-propolis-cinnamon essential oil polymeric system, cell viability was better compared to the results obtained in the case of keratinocytes cultured in the presence of textiles impregnated with the polymeric system based on xanthan-propolis-cinnamon essential oil. Also, a moderate increase in nitric oxide (**Fig. 2.b.**) can be observed in the case of textiles impregnated with the polymeric system based on chitosan.




a)




b)

***Fig. 2:*** *Graphical represenatations of: a - Cell viability test - MTT after 5 hours of incubation; b - Relative level of NO released in culture medium in two-dimensional system*

## 5. CONCLUSIONS

Two types of blended textile materials with different fiber composition impregnated with emulsions based on chitosan-propolis-cinamon essential oil and xanthan-propolis-cinamon essential oil were assessed in order to explore their physical-mechanical, physical-chemical, antimicrobial and biological properties and to find out the possible applications in developing bioactive dressings destined for the treatment of inflamatory skin injuries. The functionalization treatments have increased the mass characteristics per unit area due to the polymeric products immobilized on the surface of the textile supports. Air and water vapour permeability of functionalized samples registers much lower scales compared to untreated ones, indicating a decrease in comfort for all selected experimental variants

Textile materials treated with both bioactive polymeric systems have shown antibacterial activity for both gram positive bacteria (*S. aureus*) and gram negative bacteria (*E. coli*) test strains. The *in vitro* biocompatibility evaluation on human skin cells confirmed the absence of cytotoxicity after the short-term exposure. As our findings showed their good biocompatibility, these newly biofunctional textiles obtained by treatment with chitosan/xanthan-propolis-cinnamon essential oil polymeric systems can become a suitable candidate for the curative treatment of skin injuries, providing no skin irritations.

## ACKNOWLEDGEMENT

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