Multifunctional modification of viscose fiber using plant extracts formulations

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Abstract. Some plant extracts with specific functions have been used to integrate with textiles, which can not only endowing the fabric with certain functions, but also accord with the concept of environmental protection and sustainable development. In this paper, codonopsis powder and thyme essential oils microcapsule were added to viscose spinning fluid. The obtained viscose fiber with various functions, such as antibacterial activity, anti-mould activity and anti-mites activity. In addition, the durability are also more than 20 washes. The multifunctional viscose fiber has a more extensive application prospect.

1. Introduction

Regenerated cellulose fibers are produced using various functional agents to have more functional. In recent years, regenerated cellulose fiber has been used in various applications, including clothing, medical treatment, industrial materials, and composites. As we know that viscose fibers belong to the regenerated cellulose fibers. The viscose nonwoven fabrics modified by durable graphene oxide was used for wearable electronics showing the potential applications as physical sensors for compression and bending [1]. Viscose fiber was combined with protein to have the action in self-heating [2]. Photoluminescent viscose fabric modified by lanthanide metal-organic framework was used for self-cleaning [3]. Due to rich nutrients, it is vulnerability to bacteria, fungi and easy to breed mites during use and storage negatively affects user and textile. Therefore, the fabrics with the function of anti-microbial properties and anti-mite properties has become an urgent need.

However, plant extracts is few as functional agents to use on multifunctional viscose fiber, which is limited in the field of application for the multi-functional viscose textile. It is common that the antibacterial effects can be achieved by durable antimicrobial finishing of the textile using antimicrobial agents or by incorporating the antimicrobial agents into viscose spinning fluid in the procedure of extrusion. For example, the antimicrobial viscose fabric prepared with subsequent deposition of silver and copper ions under the effect of dielectric barrier discharge [4]. Silver nanoparticles and ions are known for their high antimicrobial activity. There is a continuing uncertainties on the impact of silver ions for humans and eco-systems. Meanwhile, metal ion antibacterial agents and chemical modification with a cross-linking agent have a certain amount of pollution to the environment in terms of manufacturing and recycling. The treatment of textile materials with plant functional agents and viscose spinning process open a possibilities to obtain effective multi-functional textile. Natural plant ingredients are never ending natural resources, environmentally-friendly while functioning well. The main source of natural and synthetic reagents on earth is plant, which has a complex structure of small molecular organic compounds and changes
significantly according to plant species and separation processes. The essence of functional properties is secondary metabolites in plant active ingredients. The effective constituents in plant extracts are excellent environmental friendly and renewable functional agents. The roots of codonopsis pilosula is used for strengthening the physique, invigorating the spleen, and nourishing the lung. It have medicinal properties from various bioactive compounds [5]. Thyme essential oil nanoemulsions have antifungal and mycotoxin inhibitory activities. And antimicrobial function studies showed that the thyme oil-loaded microcapsules are more effective than thyme oil for microorganisms. Thyme has potential as a biopesticide against stored product pests in practical application. Meanwhile, thyme essential oil also have the highly acaricidal ability and can be used for ticks control. They are used against a broad range of microorganisms of fungi and bacteria such as gram-positive bacteria Staphylococcus aureus (S. aureus), gram-negative bacteria Escherichia coli (E. coli), Candida albicans (C. albicans), Aspergillus niger (A. niger), etc. And thyme essential oil was also used against dust mites [6-8].

Herein, we report the present work about using codonopsis extraction fluid and thyme essential oils microcapsule for the preparation of multi-functional viscose fiber (MVF). We effectively combine various functional plant extracts with viscose macromolecules. The treated multi-functional viscose fabric is characterized by SEM analysis, mechanical performance testing and tested by antibacterial, anti-mould and anti-mites experiments. Our work retains and enriches the long functionalities of viscose fabric without contamination and poisoning by simple production process.

2. Materials and methods

2.1. Materials

Codonopsis was kindly supplied by LinCao science Inc. China. Thyme essential oils microcapsule (O / W, D90 = 1.611 μm, 90 % of microcapsules with a diameter of 1.611 microns) and bacterial strains were obtained from FuChi science Inc. China. And the encapsulation efficiency of thyme essential oils microcapsule was 40%. Viscose spinning solution (8.9% cellulose) was purchased from YinYing science Inc. China. All kinds of culture mediums were purchased from Land Bridge technology Co., LTD., China. All other chemicals used were of laboratory grade and were obtained from China National Pharmaceutical Group Chemical Testing Co., Ltd., such as concentrated sulfuric acid (98 w/w %), NaSO₄ (≥ 99 w/w %), ZnSO₄·7H₂O (≥ 99.5 w/w %), NaOH (≥ 96 w/w %).

2.2. The production of multi-functional viscose fiber (MVF)

Extraction processing of codonopsis extraction fluid was by means of ultrasonic extraction. The pieces of codonopsis with methanol (75 w/w %, liquid solid ratio of 15:1.) was carried out twice in the ultrasound environment for 60 minutes. The concentrated solution was further filtered and dried to obtain the plant extracts powder of codonopsis. Functional agents was made of codonopsis extraction fluid (20 wt %) and thyme essential oils microcapsule (2 v/v %). Spinning bath was made of 230 g of NaSO₄ and 15 g of ZnSO₄ mixed in 1 L of distilled water with 110 g of concentrated sulfuric acid. 5 g of NaOH was dissolved in 1 L distilled water to be the alkali lotion. The steps of viscose fiber production was schematically depicted in Scheme. Functional agents and viscose spinning solution were combined to be the multi-functional viscose spinning solution, then stirred at 750 r/min for 50 min to eliminate air bubbles and to ensure that the mixture was thoroughly mixed. The functional agents was dissolved in spinning fluid and the solution was extruded through spinneret holes to be the fiber form in spinning bath at 50-60 °C. And followed the neonatal fibers were stretched in roller drafting and then immersed in the alkali lotion. Then the fiber thus obtained was washed and dried. The schematic presentation of instrument parameters and production process was presented in Fig. 1.
2.3. Characteristics of multi-functional viscose fiber (MVF)

The surface morphology of ordinary viscose fiber (OVF) and MVF was studied by scanning electron microscopy (SEM, Phenom ProX, Holland).

2.4. Experiment of Multi-functional Viscose fiber (MVF)

2.4.1. Mechanical properties. In order to estimate the fracture strength and fracture elongation of two kinds of viscose fibers was measured using China national standard (GB/T 14337-2008) on LLY-06E Electronic single fiber strength meter.

2.4.2. Antibacterial experiments. The antibacterial activity of multi-functional viscose nonwoven fabric was quantitatively evaluated by China national standard (GB/T 20944.3-2008) with shake flask method.

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\text{Antibacterial activity(\%) = } \left( \frac{W_t - Q_t}{W_t} \right) \times 100
\]

Where \( W_t \) (CFU/mL) is average concentration of live bacteria with three control samples in shaking flask after 18h and \( Q_t \) (CFU/mL) is average concentration of live bacteria with three samples tested in shaking flask after 18h.

2.4.3. Anti-mould experiments. The anti-mould activity of multi-functional viscose nonwoven fabric was quantitatively evaluated by China national standard (GB/T 24346-2009) with petri dish test method.

2.4.4. Anti-mites experiments. The mites used in the anti-mites test are dust mites. The anti-mites activity of two viscose nonwoven fabric was quantitatively evaluated by China national standard (GB/T 24253-2009) with expulsion test method.

3. Results and discussion

3.1. Characterization of multi-functional viscose fiber (MVF)

Due to the addition of functional agents, the color of MVF is different from OVF. SEM images (Fig. 2) clearly indicate surface deposition of residual functional agents on viscose fiber. Compared the surface of OVF, MVF has many tiny particles with many long lengthways grooves. These tiny functional agents particles may increase the functional stability and washing resistance of MVF.
3.2. Mechanism of multi-functional viscose fiber (MVF)

The mechanical properties of OVF and MVF are shown in Table 1. It shows the results of fracture strength and fracture elongation of viscose fiber treated with two conditions of dry and wet. The fiber fracture strength decreased with adding functional agents slightly. On the other hand, the fiber fracture elongation increased with adding functional agents slightly. Our results are conformed to the law of change with performance of viscose fiber.

|                | Fracture Strength (cN / dtex) | Fracture Elongation (%) |
|----------------|-------------------------------|-------------------------|
|                | Dry  | Wet  | Dry  | Wet  |
| OVF            | 2.38 | 1.62 | 18.31| 24.29|
| MVF            | 2.12 | 1.52 | 19.83| 25.35|

3.3. Functional properties of multi-functional viscose fiber (MVF)

The antibacterial activity was calculated by each sample in Table 2. The results of each unwashed sample, after 10 washing cycles and after 20 washing cycles against A. niger activity were evaluated in Table 3. The results of each unwashed sample and after 20 washing cycles against dust mites activity were evaluated in Table 4.

|                | Bacterial reduction-unwash (%) | Bacterial reduction- after 20 wash(%) |
|----------------|-------------------------------|--------------------------------------|
|                | S. aureus | E. coli | C. albicans | S. aureus | E. coli | C. albicans |
| OVF            | 0         | 0       | 0           | 0         | 0       | 0           |
| MVF (without lobetyolin) | 95 ± 0.5 | 99 ± 0.12 | 79 ± 0.35 | 96 ± 0.7 | 91 ± 0.53 | 80 ± 0.43 |
| MVF            | 99 ± 0.5 | 99 ± 0.12 | 92 ± 0.35 | 98 ± 0.7 | 98 ± 0.53 | 90 ± 0.43 |

|                | Unwashed sample | Sample-after 10 wash | Sample-after 20 wash |
|----------------|-----------------|----------------------|---------------------|
| OVF            | 4               | 4                    | 4                   |
| MVF            | 0               | 0                    | 0                   |
| Control group  | 4               | 4                    | 4                   |
| Blank control group | 4           | 0                    | 0                   |
Table 4. Test results on anti-mites activity of two non-woven fabrics of viscose fibers

|                          | OVF          | MVF          |
|--------------------------|--------------|--------------|
| Cultivation time         | 24 h         | 24 h         |
| Number of dust mites in control group | 106          | 104          |
| Number of dust mites in test group | 106          | 25           |
| Reduction of dust mites (%) | 0            | 76.53        |
| Evaluate                 | Not anti-mite| Anti-mite    |

In case of antibacterial properties, the efficacy got reduced about bacteria and fungi needed to reach the value of more than 70% against S. aureus and E. coli, and 60% against C. albicans respectively. As evident from the results, multi-functional viscose showed good antibacterial activity and resistance against washing resulting in the durability of antibacterial properties till 20 washes. Moreover, Table 3 showed the addition of lobetyolin enhanced antibacterial activity. Moreover, Table 3 and Table 4 showed the anti-mould and anti-mites effects of multi-functional viscose were excellent. The ingredients contained in codonopsis have extremely antibacterial effects. Thymol was phenolic compounds of the major antimicrobial components in thyme essential oils. It exhibited the strongest antifungal efficacy on inhibition of mycelium growth. Thyme oil also had strong inhibitory activity against the S. aureus and E. coli. And thyme essential oils effected on C. albicans germ tube formation, metabolism, and biofilm disruption. Meanwhile, thyme essential oils is a kind of pure plant acaricidal agents as an ovicidal, larvicidal, and adulticidal activity against dust mites.

4. Conclusion

Hence, our study is based on adding functional agents in viscose spinning solution to prepare a new composite multi-functional viscose fiber (MVF) by the wet spinning method. It has good blending that following the functional agents and viscose have no phenomenon of interface separation. A binary combination of codonopsis and thyme showed synergistic activity, due to the combined activities of two or more components of plant extract. Hence, even minor constituents may be potentially very active in combination at low concentrations, and even the major constituents of plant extract assist a variety of mechanisms to enhance functional effects. Compared with OVF, MVF had splendid antibacterial activity, anti-mould activity and anti-mites activity, similar breaking strength and higher elongation-at-break, which meets the requirement of textile fibers. Therefore, MVFs are promising materials for functional clothing applications.

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