습식방사를 이용한 silk fibroin/silver nanoparticles 복합섬유의 제조

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Preparation of Silk Fibroin/silver Nanopaticles Wet Spun Filaments

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1. Introduction

Silk fibroin is a protein fiber that can be applied to biotechnical and bioactive polymer with mechanical strength, elasticity, and suppleness [1]. Due to biocompatibility and biodegradability, regenerated silk fibroin have been proposed for many purposes with various forms such as sponge, membrane and film [2]. Silver has a long history of successful medical and public health use and has been claimed to kill many different disease organisms due to its nano-size and larger total surface area per unit volume. Moreover, silver is skin friendly and does not cause skin irritation [3]. In this study, we prepared silk fibroin/silver nanoparticles filaments by the wet-spinning process.

2. Experimental

2.1. Materials

Silk fabrics were purchased from Korea Apparel Testing & Research Institute (KATRI) and all chemicals were acquired from Sigma-Aldrich Co., Ltd. as analysis grade and used without further purification. To insure the elimination of sericin, silk fabrics were degummed by boiling in 0.5% Na_2CO_3 solution for 1 hour and it was washed with distilled water. The degummed silk fabrics were dissolved in mixture of solvents composed of $CaCl_2/C_2H_5OH(EtOH)/H_2O$ (1:2:8 in molar ratio) at 95℃ for 5 hours and dialyzed in a cellulose membrane tube (Nominal MWCO:12,000-14,000) against distilled water for 3 days at room temperature to remove salts. After filtering, this aqueous SF solution was freezed at -74℃ for 2 days and dried at -41℃ for 3 days to obtain the regenerated SF sponge. The nano-silver colloid (SNSW, nano-silver ethanol based colloid) was supplied from NP-Tech Co., Ltd., Korea for this study.

2.2. Preparation of silk fibroin/silver nanoparticles filaments

To prepare the silk fibroin dope solution, the regenerated silk fibroin sponge were dissolved in 98% formic acid at room temperature for 1 day. The silk dope solution was prepared with various concentrations for wet spinning and it was spun through 21-gauge syringe into a different coagulation solutions (100/0, 70/30, 50/50, 30/70, and 0/100 in methanol : SNSE ratio) with 100ppm silver concentration using a syringe pump. The silk

fibroin filaments were left in the coagulation solution overnight to complete solidification, crystallization, and adhesion of silver particles. Then, these filaments were drawn by 4~5 fold in length and they were dried at room temperature for 1 day.

2.3. Characterization

The morphology, such as crosss-section and longitudinal surface, of gold coated silk fibroin/silver nanoparticles filaments was observed by scanning electron microscope (SEM). Tensile strength and elongation at break were measured using a tesile tester (Instron) according to the test standard KSK0323. Thermal behavior of the silk fibroin/silver nanoparticles filaments was analyzed by differential scanning calorimeter (DSC). DSC measurements were performed at a heating rate of 10℃/min from 50 to 400℃ under nitrogen gas. The antibacterial efficacy of the silk fibroin/silver nanoparticles filaments was quantitatively evaluated according to the test method AATCC 100-2004.

3. Results and discussions

The viscosity of silk fibroin dope solution has a great influence on formation of wet-spun filaments. In case of the silk fibroin dope solution having the low viscosity, we couldn't produce fibers. The morphology of silk fibroin/silver nanoparticles filaments were shown by SEM images, and they had very fine and smooth surfaces and circular cross-sections with micron diameter. According to measurement of particular tensile strength, the silk fibroin/silver nanoparticles filaments exhibited excellent mechanical properties. The result of antibacerial efficacy of silk fibroin/silver nanoparticles filaments was indicated good bacterial reductions, 99.9% against *S. aureus* and *K. pneumoniae.*

4. Conclusions

We prepared silk fibroin/silver nanoparticles filaments by the wet-spinning process. Although these filaments had a diameter of micron scale, they exhibited excellent mechanical properties from SEM and tensile strength analysis. Also, these filaments had excellent antibacterial efficacy against *S. aureus* and *K. pneumoniae.* Therefore, silk fibroin/silver nanoparticles filaments have great potential for the medical applications.

References

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