Preparation of Silk Fibroin/silver Nanoparticles 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 sercin, silk fabrics were degummed by boiling in 0.5% Na2CO3 solution for 1 hour and it was washed with distilled water. The degummed silk fabrics were dissolved in mixture of solvents composed of CaCl2/C3H6OH(EtOH)/H2O (1:2:8 in molar ratio) at 95°C 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 freeze at -74°C for 2 days and dried at -41°C 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 cross-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 tensile 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°C/min from 50 to 400°C 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 antibacterial 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