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Effect of degumming on structure and mechanical properties of silk textile made with silk/polyurethane core-spun yarn

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Abstract

Although silk textile shows excellent performance when used in clothing over a long period, its limited elongation and elasticity have restricted its extension to other textile and non-textile applications. In the present study, silk textile was produced using silk/polyurethane core-spun yarn and degummed to enhance its elongation and elasticity. The effects of degumming on the structure and mechanical properties of the silk textile were examined. Scanning electron microscopy observation revealed that the silk filaments became finer and more flexible with degumming, resulting in increased tangling of weft yarns and a highly shrunk textile structure in the weft direction. Although the strength of the degummed silk textile was decreased, its elongation greatly increased by 383% (a 16-fold increase) because of the degumming treatment. In particular, the elasticity of the silk textile was greatly improved. The silk textile exhibited ~30% reduction in the elongation after the second extension; however, the elongation almost did not change after 18 additional extension–recovery tests.

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Introduction

Silk is considered the best textile because of its excellent luster and feel when used in textile applications. Recently, silk has been used as a promising biomedical material including as a nerve guidance conduit (Park *et al.*, 2015), artificial bone substitute (kweon *et al.*, 2011), artificial ear drum (Kim *et al.*, 2010), and membrane for guided bone regeneration (Song *et al.*, 2011) because of its good biocompatibility (Sakabe *et al.*, 1989; Um *et al.*, 2002). However, the limited elongation and elasticity of silk textile have restricted its use in other textile and non-textile applications. In a previous study, as a preliminary work, silk textile was made with silk/polyurethane core-spun yarn and treated with hot water to improve its elongation and elasticity (Bae and Um, 2016). However, the results were unpromising, with the silk textile exhibiting 96.7% elasticity, even although it was just extended by an elongation of 20%.

In the present study, the silk textile made with silk/polyurethane core-spun yarn was degummed to enhance its elasticity, and the effects of degumming on the structure and mechanical properties of the silk textile were examined.

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Materials and Methods

Preparation

A silk/polyurethane core-spun yarn consisting of two silk yarns (sheath) made by twisting four 21-denier silk filaments around a polyurethane filament (core) was used as the weft yarn, and two 42-denier silk yarns were used as the warp yarn to weave the silk textile. The degree of twist of the weft yarn was 4000 turns per inch. The silk textile was degummed to remove the sericin and then dried at room temperature.

Measurement and characterization

The morphologies of the silk textiles were examined using field-emission electron microscopy (FE-SEM; S-4800, Hitachi, Japan). The samples were coated with Pt–Pd before FE-SEM imaging. The shrinkage of the silk textile after degumming treatment was calculated using eq. 1 (Bae and Um, 2016):

$$\text{Shrinkage (\%)} = (L_1\text{-}L_2)/L_1 \times 100 \qquad \text{(eq. 1)}$$

L_1: Length of textile before degumming treatment
L_2: Length of textile after degumming treatment.

A Fourier-transform infrared (FTIR, Nicolet 380, Thermo Fisher Scientific, USA) spectrometer in the attenuated total reflection mode was used to examine the molecular conformation and crystallinity index of the silk textiles. The crystallinity index was calculated from the FTIR spectrum as the intensity ratios of 1260 and 1230 cm^{-1} using eq. 2:

$$\text{Crystallinity index (\%)} = A_{1260\ cm^{-1}} / A_{1230\ cm^{-1}} \times 100 \qquad \text{(eq. 2)}$$

$A_{1230\ cm^{-1}}$: Absorbance at 1230 cm^{-1}
$A_{1260\ cm^{-1}}$: Absorbance at 1260 cm^{-1}

Stress–strain curves were obtained using a universal test machine (UTM; OTT-03, Oriental TM, South Korea) to evaluate the mechanical properties of the silk textiles. The tensile tests were performed using a 200-kgf load cell at an extension rate of 100 mm/min for the silk textile. The silk textiles were cut into 70 mm × 25 mm pieces. The gauge length was 30 mm. All the samples were preconditioned at 20°C and 65% (relative humidity); five samples for each condition were tested, and the average and standard deviation of the measurement results were used.

The test method used for the elastic recovery of the silk textiles was introduced in a previous report (Bae and Um, 2016). Briefly, silk samples with the same dimensions were extended to 10 mm at an extension rate of 1 mm/s. Before the extension, the boundary line between the clamps was marked. The silk textile in the clamps remained extended for 1 min after extension. The clamps were then removed from the silk textile to allow it to recover to its original state (i.e., the state before extension). The elastic recovery ratio was calculated using eq. 3.

$$\text{Elastic recovery ratio (\%)} = (10 - Y)/(10 \times 200) \qquad \text{(eq. 3)}$$

Y (mm) = X – 50 mm
X (mm) = length between the marked boundary lines

Repetitive extension tests were applied to the silk textile using the UTM. The gauge length of the silk textile (30 mm) was extended to 90 mm (200% elongation), and then the textile was allowed to recover to its original gauge length. The extension and recovery test for the silk textile was repeated 20 times. The extension rate was 500 mm/min, and the other conditions (sample size, load cell, and temperature/humidity) were the same as those used for the stress–strain curve test.

Results and Discussion

Morphology and shrinkage of silk textile

Fig. 1 presents an SEM image of the silk/polyurethane core-spun yarn used as the weft yarn. The polyurethane filaments were located on the core side, and the silk filaments covered the polyurethane filaments. Because the sericin was removed by the degumming process, the silk filaments were separated into many silk fibroin fibers.

To examine the effect of degumming on the morphology of the silk textile, FE-SEM was used, and the results are presented in Fig. 2. As the silk textile was degummed, the weft yarn became very tangled, leading to shortening of the distance between warp yarns. Finally, substantial shrinkage of the silk textile was observed in the weft direction. The shrinkage rate of the silk textile in the weft direction was 63.6%.

A similar phenomenon was previously observed for silk textile made with silk/polyurethane core-spun yarn (Bae

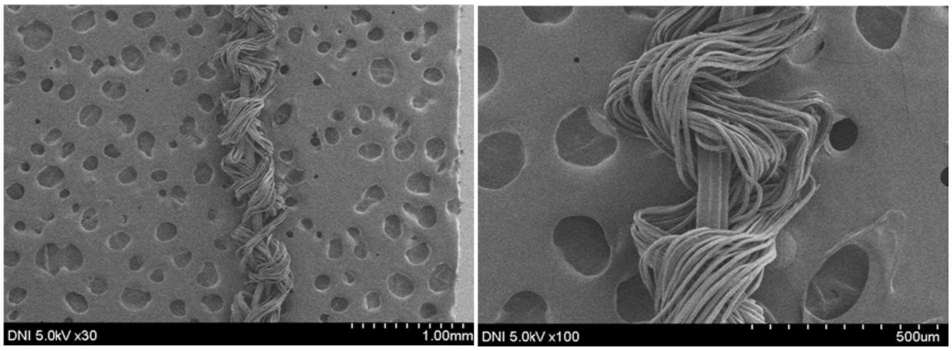

Fig. 1. FE-SEM image of degummed silk/polyurethane core-spun yarn.

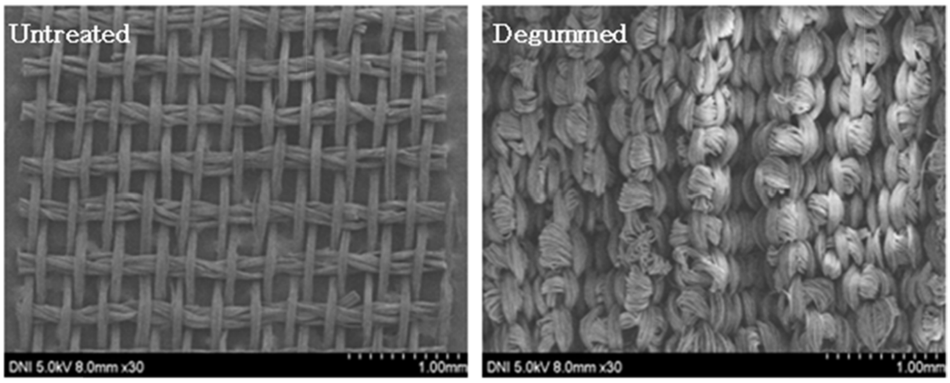

Fig. 2. Effect of degumming on the morphology of silk textile made with silk/polyurethane core-spun yarn.

and Um, 2016). That is, the weft yarn was tangled by water treatment at high temperature (100°C), leading to shrinkage of the silk textile. However, the degumming process resulted in a more tangled yarn morphology than the water treatment at high temperature. Although the exact reason cannot be elucidated in this study, it is assumed to be related to the decrease of the silk filament diameter by degumming. As the sericin is removed from the silk filament by degumming, the silk filament becomes finer because one silk filament is transformed into two silk fibroin filaments. As the result, the finer silk fibroin filament becomes more flexible and can be more tangled than that exposed to the hot water treatment because the sericin cannot be fully removed by hot water and the thicker silk filament is less flexible for tangling.

Structural characteristics and mechanical properties of silk textile

The molecular conformation of the silk is examined using FTIR spectroscopy because it strongly affects the mechanical properties of silk (Chung *et al*., 2015; Kim and Um, 2014; Um *et al*., 2001). Therefore, the effect of degumming on the molecular conformation of silk textile was examined using FTIR spectroscopy, and the results are presented in Fig. 3(A). All the silk textiles exhibited IR absorption peaks at 1620, 1515, and 1260 cm^{-1}, representing amide I, II, and III bands, respectively. These peaks are attributed to β-sheet conformation, indicating that the silk textiles exhibit β-sheet conformation. The IR peak at 1260 cm^{-1} in the amide III band became more evident when the silk textile was degummed. This result indicates that the crystallinity of the silk textile increased with degumming, which can be more clearly observed in the crystallinity index result in Fig. 3(B).

Fig. 4(A) shows the effect of degumming on the stress–strain curve of the silk textile made with silk/polyurethane core-spun yarn. The untreated silk textile exhibited rigid properties, displaying a steep increase of stress. In contrast, the degummed silk textile was extended without stress until a strain of 180%. After that, the stress increased constantly until a strain of 383%. This result indicates that the degummed silk textile made with silk/polyurethane core-spun yarn is a very ductile and extensible material. As observed in Fig. 4(B) and

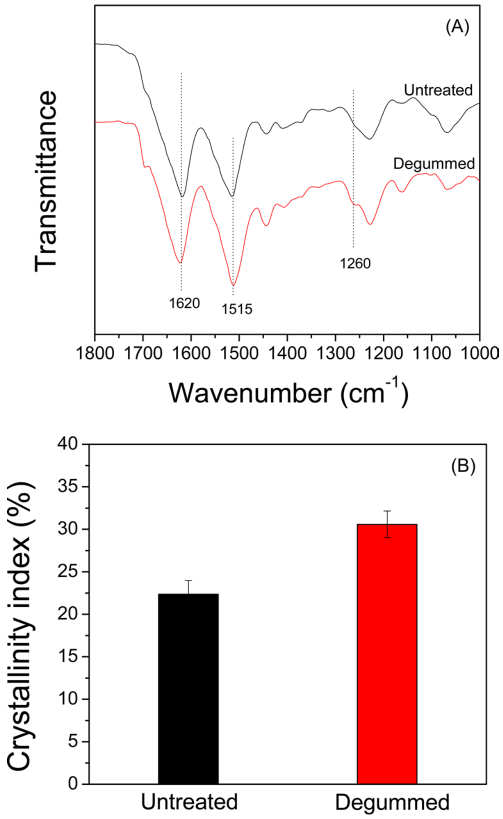

Fig. 3. Effect of degumming on (A) FTIR spectra and (B) crystallinity index of silk textile made with silk/polyurethane core-spun yarn.

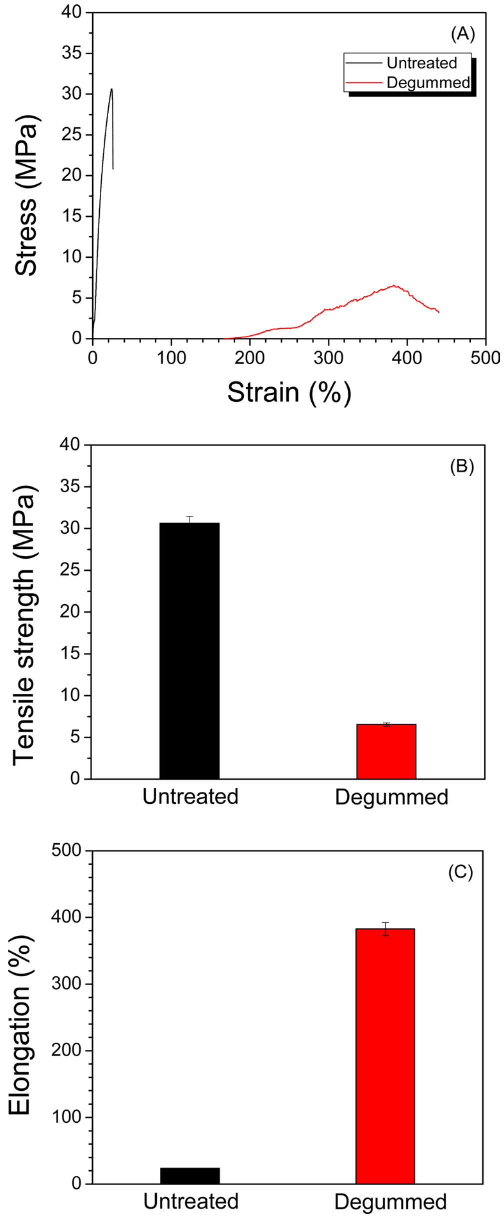

Fig. 4. Effect of degumming on (A) stress–strain curve, (B) tensile strength, and (C) elongation of silk textile made with silk/polyurethane core-spun yarn.

4(C), when the silk textile made with silk/polyurethane core-spun yarn was degummed, the tensile strength decreased from 30.6 to 6.5 MPa and the strain increased by 16 fold (24% to 383%). This result indicates that the degumming process causes the silk textile to become remarkably extensible. The tangled weft yarn becomes untangled by the tensile force, which allows the silk textile to become easily extended by the external force.

Considering that the hot-water-treated silk textile exhibited 140% elongation in the preliminary study (Bae and Um, 2016), the elongation of 383% of the degummed silk textile indicates that degumming is a more effective tool to improve the elongation of silk textile made with silk/polyurethane core-spun yarn than hot water treatment. As mentioned in the previous section, the discrepancy between the effect of the hot water treatment and that of degumming might be related to the fineness of the silk filament. That is, when the silk textile is degummed, sericin is removed from silk filament. As a result, the silk filament splits into two silk fibroin filaments, leading to finer filaments in the silk textile. The finer filament makes the silk textile more flexible and more shrinkable by degumming. The more shrunken textile and more tangled yarns can be further extended by the tensile force.

Because clothing is under repetitive external force in actual use, the elastic recovery of a textile is also important. Therefore, in the previous study, the elastic recovery ratio of the hot-water-treated silk textile was tested. The value was unsatisfactory (96.7%), considering 20% elongation was used once in the measurement (Bae and Um, 2016). However, in the present study, the elastic recovery ratio of the degummed silk textile

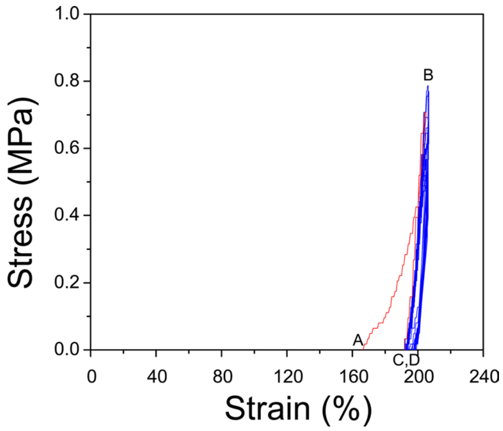

Fig. 5. Repetitive extension–recovery test result for silk textile made with silk/polyurethane core-spun yarn.

was 100%. Therefore, a tougher test method for elastic recovery properties was used in this study. That is, the silk textile was extended until an elongation of 200%, and the extension and recovery were repeated 20 times; the results are presented in Fig. 5. At the first extension (red color), the silk textile was extended to 165% elongation without stress (point A), and the stress then increased significantly until 200% (point B). When the silk textile was allowed to recover (i.e., the distance between the gauges was decreased to the original gauge length), the stress decreased to zero at an elongation of ~195% (point C). At the second extension (blue color), the silk textile was extended to an elongation of ~195% (point D). When the silk textile was allowed to recover again, the stress decreased to zero at an elongation of ~195%.

Interestingly, point A (165% elongation) for the first extension moved to point D (~195% elongation) for the second extension, and point D remained almost constant upon further extension (until the 20th extension). This result indicates that the elongation of the silk textile is reduced by ~30% with the first extension; however, it remains almost constant with further extension.

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Conclusions

In this study, a silk textile made with silk/polyurethane core-spun yarn was degummed to improve its elongation and elasticity, and the effects of degumming on the structure and mechanical properties were examined.

The elongation of the silk textile made with silk/polyurethane core-spun yarn greatly increased (16 fold) with degumming compared with the untreated silk textile. Although the elongation of the silk textile was reduced by ~30% with the first extension, the value remained almost constant upon further extension. This finding indicates that the silk textile exhibited a very high elongation and elasticity. Thus, this silk textile shows promise for extensive use in various textile and non-textile applications.

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