Structure and Improvement of Properties of Floss Silk via Scouring and Finishing Treatment

Abstract
In the present study, floss silk was treated by scouring and finishing, respectively. The microstructure was observed with scanning electron microscopy (SEM), the mechanical property tested by an Instron 5566 tensile tester, and the crystal structure was analysed with Fourier transform attenuated total reflection infrared spectroscopy (FTIR) and X-ray diffraction (XRD), respectively. The results show that the properties of floss silk treated with the refining method are better than with the alkali method.

Key words: floss silk, finishing treatment, structure, silk fibre properties.

Introduction
Floss silk is improved from native silk, which includes excellent properties such as soft, suitable and excellent moisture absorbency. Garments made from floss silk are high-price and offer health-protection. A quilt made from floss silk can protect the skin and cardiovascular system. Floss silk is an important development of native silk and has been studied by many researchers. Maingot et al. studied the lattice posterior operation for a direct inguinal hernia of floss silk [1]; Ramadan et al. employed dental floss silk to transient bacteremia [2]; Zhao et al. studied the biological characteristics of floss silk [3]; Chen et al. researched the optimum design of processing conditions for differential tussah/Mulberry elastic floss silk [4]; Chu et al. developed some multifunction silk floss quilts [5], and Maingot researched the floss silk lattice for unguinal hernias [6].

Scouring is a very important process for floss silk manufacturing as it removes natural impurities and weaving slurry. Scouring includes three steps: firstly, the Na2CO3 solution (wt% = 0.04 g/dm³), H2O2 (wt% = 10 mL/dm³), textile soap 920 (wt% = 0.5 g/dm³) and degreaser solution TF-101BN (wt% = 1 g/dm³) were mixed; samples were put into the solution for 1 hour (Na2CO3:water = 1:100), then dried and conditioned at conventional conditions (temperature 20 °C and relative humidity 60%), and finally dried at 100 - 105 °C. Textile soap 920 was provided by the Baode chemical factory, Shanghai, China. Degreaser solution TF-101BN was provided by Chuanhua Chemical Co. Ltd., Zhejiang, China. Scoured solution C-180 was provided by Baode Chemical factory. Soft agent BG1-311 and TF-404A were both provided by Baode Chemical factory.

Experimental methods
Alkali degumming processing
The samples were washed in Na2CO3 solution for 1 hour (Na2CO3:water = 1:100 and temperature 100 °C), then dried and conditioned at conventional conditions (temperature 20 °C and relative humidity 60%), and finally dried at 100 - 105 °C.

Scoured agent degumming processing
Scoured agent degumming processing includes three steps: firstly, the Na2CO3 solution (wt% = 0.04 g/dm³), textile soap 920 (wt% = 1 g/dm³) and degreaser solution TF-101BN (wt% = 1 g/dm³) were mixed; samples were put into the solution and boiled for 45 min, then they were washed 3 times with deionised water of 40 °C. Secondly textile soap 920 (wt% = 1 g/dm³), soft agent BG1-311 (wt% = 0.5 g/dm³) and scouring agent C-180 (wt% = 0.04 g/dm³) were mixed; samples from step 1 were put into the solution and boiled for 60 min; then they were washed 3 times with deionised water of 40 °C and finally dried at 100 - 105 °C. Thirdly the samples from step 2
were put into the soft solution TF-404A (wt% = 10 g/L) for 60 min; then their pH was improved by about 7 via glacial acetic acid, and next the samples were washed 3 times with deionized water of 40 °C and finally dried at 100 - 105 °C.

Floss silk SEM analysis
Scanning electron microscopy analysis was carried out on a field emission SEM (Quanta 200, manufactured by FEI, Holland), with the samples being coated with gold before testing.

Floss silk mechanical property analysis
The mechanical properties of samples were tested with an Instron 5566 Universal Tensile Tester (Great Britain), at a gauge length of 10 cm and strain rate of 50 mm/min. The width of the sample was 15 cm × 5 cm. Samples were tested 5 times and the results averaged.

Floss silk FT-IR analysis
The groups and basic changes of floss silk treated and untreated were observed by an Infrared Reflectoscope Reflector (TENSOR 27, manufactured by BRUKER, Germany).

Floss silk XRD analysis
Wide-angle X-ray diffraction (WAXD) analysis of the floss silk was carried out on an X-ray diffractometer (D/MAX-1200; Rigaku Denki, Tokyo, Japan) by the reflection method using a CuKa target at 40 kV and 30 mA. The diffraction angle ranged between 4° and 40°. All samples were cut to a particle-like size to erase the effect of the crystalline orientation of each sample. The integrated peak intensity for each high crystal reflection selected and the amorphous background were extracted by a curve-fitting program with the multiple peak separation method. The crystallinity (vc) was obtained from the ratio of the integrated area of all crystal peaks to the total integrated area (including the amorphous area) according to the peak fit results.

Results and discussion
Surface morphology analysis
Figure 1 shows the surface morphology of floss silk fibres under various treating methods. Figure 1.a is the original across morphology of floss silk fibre. Figures 1.b to Figure 1.d shows the across morphology of floss silk fibre treated with Na₂CO₃ - wt% = 0.05 g/dm³ (b, g), Na₂CO₃ - wt% = 5 g/dm³ (c, h), Na₂CO₃ - wt% = 10 g/dm³ (d, i), and scouring agent degumming (e, j).

Figure 1. Across (a - e) and longitudinal (f - j) morphology of floss silk fibres: original (a, f), treated with Na₂CO₃ - wt% = 0.05 g/dm³ (b, g), Na₂CO₃ - wt% = 5 g/dm³ (c, h), Na₂CO₃ - wt% = 10 g/dm³ (d, i), and scouring agent degumming (e, j).
Table 1. The mechanical properties of floss silk fibres.

<table>
<thead>
<tr>
<th>Treated methods</th>
<th>Untreated</th>
<th>treated with Na$_2$CO$_3$, g/dm$^3$</th>
<th>Scoured agent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Strength, cN</td>
<td>0.05</td>
<td>5.0</td>
</tr>
<tr>
<td></td>
<td>11170 ± 320</td>
<td>12130 ± 310</td>
<td>10270 ± 330</td>
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</table>

Fibre XRD analysis

XRD analysis was used to test the change in crystal structure of floss silk before and after scouring/finishing treatment, as shown in Figure 2.b. There is a strong peak for Silk II at 26.0° and two weaker peaks at 9.2° and 24.5°. However, there is no obvious influence on the molecular conformation of floss silk fibre.
be calculated using PeakFit software; the crystallisation degree of the four curves is 42.44%, 43.34%, 40.88% and 41.31%, respectively. The results indicate that the alkali degumming process and scouring agent degumming process have no obvious influence on the crystal structure of floss silk fibre.

Conclusions

The alkali degumming process and scouring agent degumming process were applied to improve the surface properties and structure of floss silk. The results show that alkali degumming processing and scouring agent degumming processing both destroy the surface of floss silk fibres; however, the fibre surface becomes much smoother after the scouring agent degumming treatment. The mechanical tests show that floss silk fibres have no obvious changes under various conditions. The FT-IR and XRD analysis results indicate that alkali degumming processing and scouring agent degumming processing both have no obvious influence on the chemical composition and crystal structure of floss silk fibres. The research results show that scouring agent degumming processing is a much better treatment to improve the surface properties and structure of floss silk fibres.

References