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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 micro structure 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. Finishing is also an extremely important process method to enhance the colour and shape effect. The exterior and hand feel of floss silk fabric can be obviously improved after scouring and finishing processing. At the same time, more special functions of fabrics can also be got. We know that the surface and mechanical properties of floss silk fabric can be obviously influenced during scouring and finishing processing. No longer is the needle point method often employed to manufacture floss silk fabric. However, there is the problem of how to control the mechanical properties of floss silk fabric in the finishing process. Therefore the surface and mechanical properties of floss silk fabric during the scouring and finishing processes are very important. However, how to improve the surface properties and structure of floss silk in the scouring and finishing is still beyond our scope. In this study, floss silk was treated by scouring and finishing under different conditions, respectively. Then the micro structure was observed with SEM (scanning electron microscopy) and the crystal structure analyzed with FTIR (Fourier transform attenuated total reflection infrared spectroscopy) and XRD (X-ray diffraction), respectively. This research can help us improve the structure and surface properties of floss silk with suitable treatment in the scouring and finishing processes.

Experimental

Experimental materials

Floss silk fabric was provided by Guihua silk Co. Ltd., Guangxi, China. The weight was 27.25 g/m^2 and thickness 0.04 mm. Samples for treatment were prepared in the size of 30 cm × 30 cm. All the samples were washed in deionized water, 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 Na_2CO_3 solution for 1 hour (Na_2CO_3: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 Na_2CO_3 solution (wt% = 0.04 g/dm^3), H_2O_2 (wt% = 10 mL/dm^3), textile soap 920 (wt% = 0.5 g/dm^3) and degreaser solution TF-101BN (wt% = 1 g/dm^3) 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/dm^3), soft agent BG1-311 (wt% = 0.5 g/dm^3) and scouring agent C-180 (wt% = 0.04 g/dm^3) 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 Na_2CO_3 (wt% = 0.05 g/dm^3, 5 g/dm^3 & 10 g/dm^3 respectively). We can see from

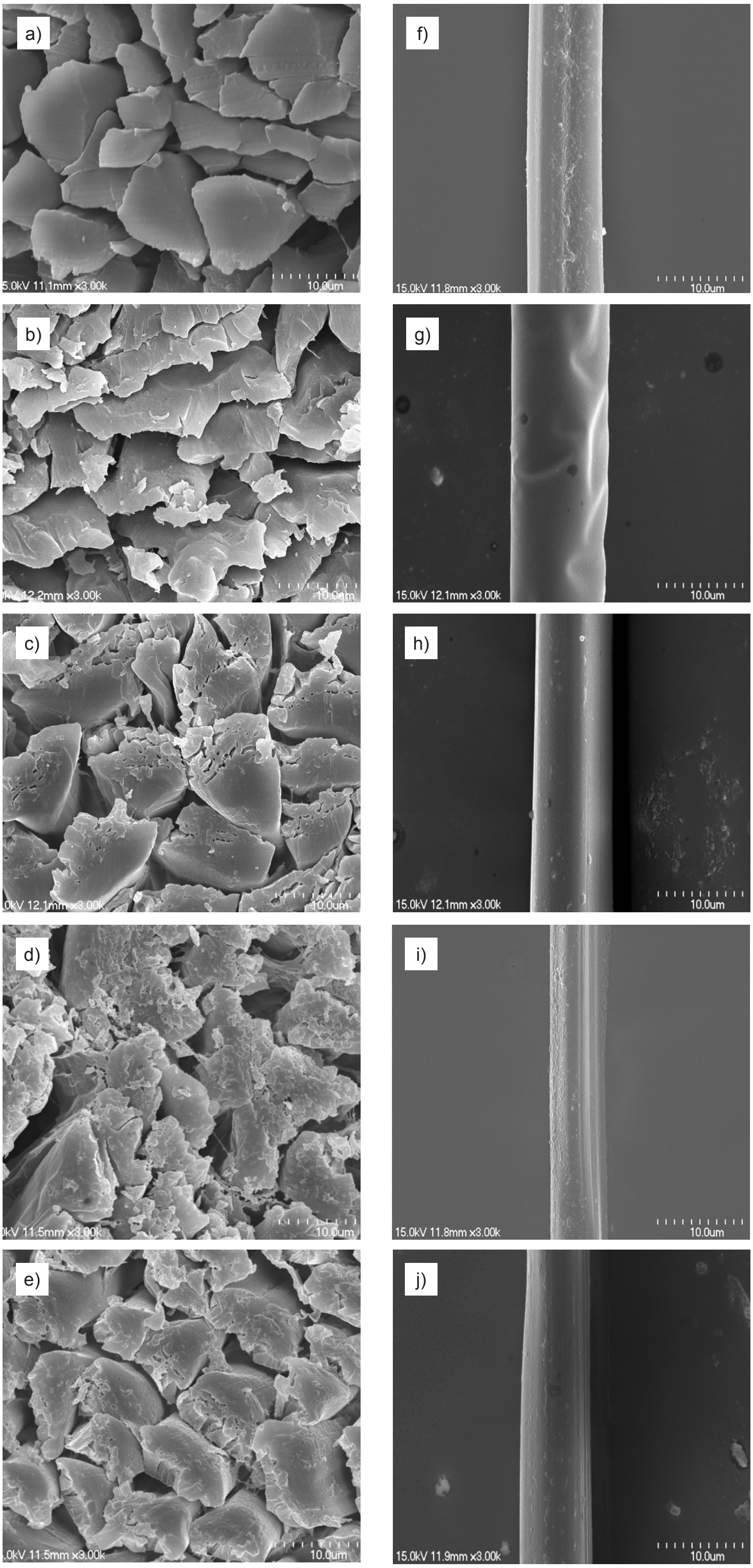

Figure 1. *Across (a - e) and longitudinal (f - j) morphology of floss silk fibres: original (a, f), treated with Na_2CO_3 - wt% = 0.05 g/dm^3 (b, g), Na_2CO_3 - wt% = 5 g/dm^3 (c, h), Na_2CO_3 - wt% = 10 g/dm^3 (d, i), and scouring agent degumming (e, j).*

Figure 1.a that the across morphology of the original fibre is smooth and looks like a triangle. The across morphology sizes of floss silk fibre treated with Na_2CO_3 (wt% = 0.05 g/dm^3) are very varied. It cannot look like a triangle because floss silk fibre is destroyed during the manufacturing process, with the across morphology also being destroyed at the same time. *Figure 1.c* shows that the across morphology of the fibre also looks like a triangle; parts of the across morphology are smooth and some interspaces appear in the across. *Figure 1.d* indicates that the across morphology is extremely serious as all of the across morphology is utterly destroyed, with some silk fibroin melts and holes also appearing at the same time. The morphology may be caused by the fact that alkali at high content melts some silk fibroin. *Figure 1.e* is the across morphology of floss silk fibre treated with scoured agent degumming processing. It can be seen that the across morphology also looks like a triangle, and parts of the across morphology are smooth. The results indicate that the across morphology of floss silk fibres will be destroyed to some degree when they are treated with alkali degumming processing and scoured agent degumming processing, respectively.

Figure 1.f is the longitudinal morphology of the original floss silk fibre; *Figures 1.g* to *Figure 1.i* show the longitudinal morphology of floss silk fibre treated with Na_2CO_3 (wt% = 0.05 g/dm^3, 5 g/dm^3 and 10 g/dm^3 respectively), and *Figure 1.j* is the longitudinal morphology of floss silk fibre treated with scoured agent degumming processing. There are many sericin spots and the surface on the original floss silk fibre is rather rough, as shown in *Figure 1.f*. It can be seen that the floss silk fibre is depressed in *Figur 1.g*, which may be because the fibres are deformed in the foot tread process. We can see that there are some spots on the surface of floss silk fibre in *Figure 1.h*, which may be residual alkali after the degumming of alkali method. In *Figure 1.i*, there are some spots and longitudinal fringes on the fibre surface; it may be that the silk fibroins are damaged at high alkali contents. We can see from *Figure 1.j* that there are also some white spots on the fibre surface, which may be the residual refining agent. In general, there are some spots in the fibre longitudinal morphology from *Figure 1.h* and *Figure 1.i*. *Figure 1.j*, which shows the longitudinal morphology is much smoother than in the original samples.

Table 1. The mechanical properties of floss silk fibres.

Treated methods	Untreated	treated with Na_2CO_3, g/dm^3			Scoured agent
		0.05	5.0	10.0	
Strength, cN	11170 ± 320	12130 ± 310	10270 ± 330	10200 ± 320	11160 ± 290

Floss silk mechanical property analysis

The mechanical properties of samples treated by various methods were tested with an Instron 5566 Universal Tensile Tester, the results of which are shown in *Table 1*. The results indicate that the mechanical properties of floss silk fibres are quite different under various treatment methods. The mechanical properties also increase when treated with Na_2CO_3 with 0.05 g/dm^3 because some friction is created on the surface of floss fibres at this condition, which increases the breaking strength. However, the tensile strength decreases when the Na_2CO_3 concentration increased, because higher Na_2CO_3 damages the physical properties of floss silk fibres. The mechanical property saw no obvious changes for the scoured agent treatment.

Floss silk FT-IR analysis

FT-IR analysis was used to test the change in chemical composition of floss silk before and after scouring/finishing treatment, as shown in *Figure 2.a*. A sharper peak is observed at 1068 and 976 for floss silk fibre treated by scoured agent degumming processing, and some other increased peaks were distributed at 1516, 1228 & 698 at the same time, which is ascribed to the oxygen-containing groups, such as s aldehyde C=O stretch. However, the FT-IR saw no obvious changes for the four samples tested, which indicates that the alkali degumming process and scouring agent degumming process have no obvious influence on the molecular conformation of floss silk fibre.

Floss silk XRD analysis

XRD analysis is used to test the change in the 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 20.6°, and two weaker peaks at 9.2° and 24.5°. However, there is spiral features diffraction peak of Silk I. The degree of crystallisation can

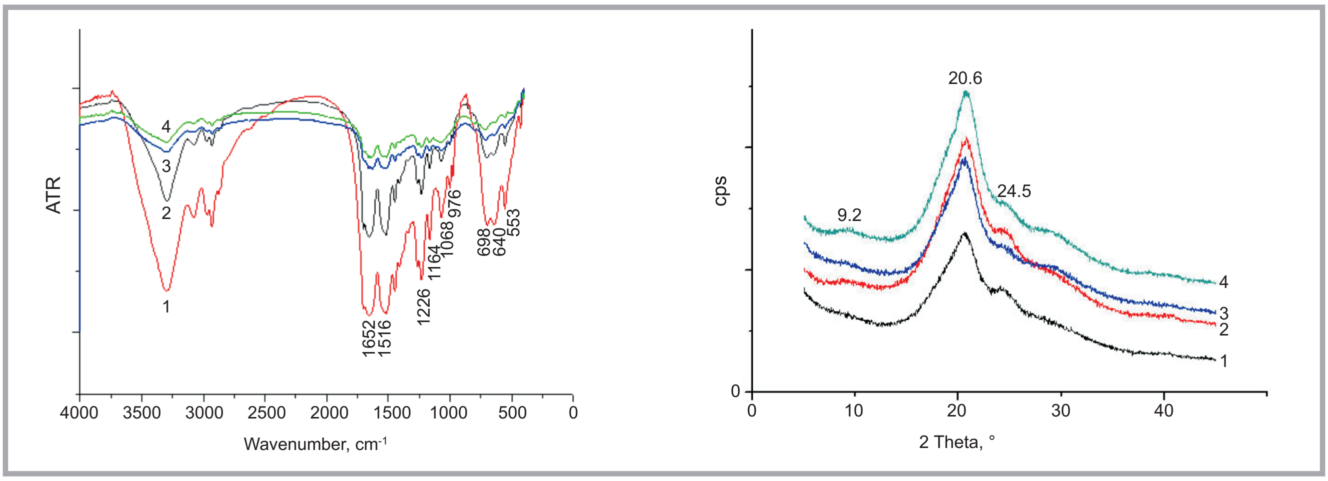

Figure 2. FT-IR (a) and XRD (b) of floss silk fibres before and after treatment at various conditions; 1 - original floss silk fibres, 2- floss silk fibres treated with scouring agent degumming, 3 - floss silk fibres treated with Na_2CO_3 (wt% = 0.05 g/dm^3), 4 - floss silk fibres treated with Na_2CO_3 (wt% = 5 g/dm^3).

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

1. Maingot R. The floss silk lattice posterior repair operation for direct inguinal hernia. *British Medical Journal* 1941; 1(4194): 777-778.
2. Ramadan A, Zaki S, Nour Z. A study of transient bacteremia following the use of dental floss silk and interdental stimulators. *Egyptian Dental Journal* 1975; 21(4): 19.
3. Zhao XP. *Biological characteristics and gardening application of pink floss-silk tree and its seed propagation*. Sugarcane, 2005.
4. Chen Y. Optimum Design of Processing Conditions for Differential Tussah/Mulberry Elastic Silk. *Journal of Textile Research* 2002; 23(1): 49-50.
5. Chu C, Sheng J, Lin H, Pan Z, Zhang L, Luo Z. Research and Development of New Multifunction Silk Floss Quilt. *Silk Monthly* 2003; 12.
6. Maingot R. The floss-silk lattice repair for unguinal hernias. *The British Journal of Clinical Practice* 1979; 33(4): 97.

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