Study on polyvinyl alcohol finishing silk

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Abstract. To improve the quality of woven embroidery products, it is necessary to improve the ability of embroidery to prevent pilling. The monofilament bundle is impregnated with 2% polyvinyl alcohol and crosslinked and cured by microwave heating at a suitable power. The finished silk samples are characterized by infrared spectroscopy and X-ray powder diffraction, scanning electron microscopy and optical microscopy. The experimental results show that after the monofilament bundle is heated by 350W microwave power for 4 minutes, the degree of aggregation of the finished monofilament bundle is greatly improved, the chromaticity of the sample is not significantly decreased, the silk feel is soft, the tensile strength is slightly enhanced, and the anti-friction ability is obviously enhanced. On this basis, further optimization of the process conditions may provide technical support for the anti-pilling finishing industrialization of products such as embroidery.

1. Introduction

With the transformation and upgrading of products, the application of embroidery has expanded from traditional costumes and decorations to many new fields such as scarves, bags and shoes, and greatly increased the added value of related products. However, the embroidery pattern is easy to fluff during friction and damage during use, which reduces the beauty and consumption experience of the product. People try to improve the fluffing performance of embroidery with a suitable finishing agent [1]. Polyvinyl alcohol (PVA) is a polyhydroxy-containing water-soluble polymer with good film forming, adhesion, emulsifying and oil resistance, excellent biocompatibility, non-toxicity, high transparency and wear resistant, so it is widely used in food, textile, agriculture, electronics and other national economic industries [2-6].

Microwave is an electromagnetic wave with a frequency between 300MHz and 300GHz. It has good penetrability and a significant heating effect on the dielectric. Because its frequency is close to the rotational vibration frequency of chemical groups, it can be used to change the molecular conformation and aggregation structure, selectively activate reactive groups and promote chemical reactions[7].

In this paper, the polyvinyl alcohol-treated silk samples were subjected to microwave irradiation, and the structural changes of silk were investigated by means of infrared, scanning electron microscopy and X-ray powder diffraction. The color, feel and single-strand tensile strength and
aggregation status of the treated silk were evaluated. This work laid the foundation for subsequent embroidery anti-pilling.

2. Experimental materials and equipment

2.1. Experimental materials
Silk, polyester (standard substrate) standard lining (Shanghai Textile Industry Technology Supervision Office), 2488 polyvinyl alcohol powder (Shanghai Building Materials Additives Company), Deionized water (homemade).

2.2. Experimental equipments
TM3030 scanning electron microscope (Hitac, Japan), Microwave oven (Guangdong Midea, EM720KG1-PW, 700W), Fourier infrared spectrometer (Thermo Fisher, iS5). CheckIII spectrophotometer in D65 light source, AGS-J1KN Shimadzu material testing machine, Fourier infrared spectrometer (Thermo Fisher, iS5).

2.3. Experimental Methods

2.3.1. Polyvinyl alcohol finishing of silk samples
A 50 cm silk single-strand sample was taken from the silk standard lining, immersed in a 2% polyvinyl alcohol solution, dried, placed in a petri dish, and taken out in a microwave oven (power P50=350W) for a few minutes, and the fabric was removed. Changes were investigated at different heating times (2 min, 3 min, 4 min, 5 min, 6 min). The change of single bundle is heated for 4 min at different powers (P50, P80=560W, P100=700W).

2.3.2. SEM observation, infrared analysis and X-ray powder diffraction characterization of silk samples.
The surface state of the single bundle before and after the treatment was observed using a TM3030 scanning electron microscope. The scanning electron microscope loading voltage was 15 kV, and the magnification was unified to 200 times.
Approximately 2 mg of silk sample was mixed with a certain amount of KBr and ground, placed in a tablet press to form a thin disc, and the thin disc was placed in a machine for testing. The test condition was a wave number scanning range of 4000 to 400 cm\(^{-1}\). The resolution is 4 cm\(^{-1}\) and the number of scans is 32.

2.3.3. Silk sample color and feel evaluation
The color properties of the silk before and after treatment were tested using the CheckIII spectrophotometer at the D65 source, the maximum absorption wavelength of 420 nm, and the USAV 6.6 mm lens aperture. Silk sample feel was examined using sensory evaluation.

2.3.4. Single bundle strength test
According to GB/T 19975-2005 "High-strength filament tensile test method" standard, single-strand wire strength was tested using AGS-J1KN Shimadzu material testing machine, monofilament fixed length 500mm, speed 500mm/ min.
3. Results and Discussion

3.1. SEM and IR spectra and X-ray powder diffraction characterization of finished silk samples

![Figure 1. Scanning electron microscope image of a single bundle of silk samples](image)

A-treated silk; B-treated silk; C-untreated silk 10 times friction; D-PVA-P50-2 min; E-PVA-P50-3 min; F-PVA-P50-4 min; G-PVA-P50-5 min; H-PVA-P50-6 min.

It can be seen from Figure 1 that the silk sample A which has not been treated with the finishing agent has a clear boundary between the single fibers in the yarn, and has a relatively clear separation state, and the surface is smooth and flat. Silk B treated with polyvinyl alcohol, the surface of the fiber in the thread adheres to the obvious film, and the single fibers which are originally separated are bonded together. The silk sample A, which has not treated with the finishing agent, cracked after being rubbed 10 times (Figure 1 C), and broken after 20 times. After the single-stranded wire B treated with polyvinyl alcohol has been rubbed 40 times, the monofilament bundle structure remains relatively intact or partially intact (D-H). Figure 1 D to Figure 1 H are SEM structures with 40 filaments rubbed by a single filament by microwave processing at different times. In 2-6 minutes, as the microwave heating time increases, the anti-friction and anti-pilling properties of the monofilament bundle are gradually enhanced. This indicates that the polyvinyl alcohol coating is beneficial to enhance the anti-friction properties of the monofilament bundle after forming a protective film on the surface of the tow and the adhesive fragile monofilament.

The infrared characteristic absorption spectrum of the protein is mainly composed of the amide I band (Amide I 1700–1600 cm⁻¹), the amide amine II band 1600–1450 cm⁻¹, and the amide III band 1200–1400cm⁻¹. It can be seen from Figure 2, that when the original silk (a) is heated by microwave, its amide I, II band has a reduced absorption intensity, and many small absorption peaks (b) appear in the large absorption band. After the polyvinyl alcohol finishing (c), the absorption peak I,II,III intensity are further weakened. It may be that the protein structure is reduced, and the random coil structure is increased. It may be that the protein regular structure is reduced, while the random coil structure increases[8].
3.2. Performance test of finished silk samples

3.2.1. Sample grayscale and softness evaluation

![Figure 2](image)

Figure 2. Infrared absorption spectrum of the sample a-pure silk, b-polyvinyl alcohol finishing silk, microwave (P50), 4 min, c-polyvinyl alcohol finishing silk, microwave (P50), 6 min.

| Power(W), 4min | Time(min), 350W(P50) |
|----------------|----------------------|
| 350(P50)       | Time(min)  | 2     | 3     | 4     | 5     |
| K/S            |       0.0639 | 0.0648 | 1.9642 | 0.0618 | 0.0622 | 0.0639 | 0.0636 |
| Grayscale      | 4-5     | 4-5   | 1     | 4-5   | 4-5   | 4-5   |

It can be seen from Table 1 that as the microwave power increases, the K/S value of the finished sample increases gradually. Within 4 minutes, the power is below P80, and the gray scale change is not significantly stable at 4-5. At the power P100, the K/S value increased sharply, the sample became yellow, and the number of stages dropped to level 1. Fixed microwave power P50, within 2-5 minutes, the K/S value and the gray scale are not changed significantly, and the degree of color change is very low at 4-5. Experiments show that under certain conditions, microwave processing of silk finished by PVA can be done without changing the color of silk [9].

3.2.2. Single bundle wire strength test after PVA treatment

| Time(min), P50 |
|----------------|
| 0      | 3      | 5      | 6      |
| Strength(N)   | 70.50  | 73.5   | 76.5   | 58.5   |
| Elongation (%) | 11.5   | 11.0   | 10.8   | 7.59   |
The tensile strength and elongation of monofilament bundles are important indicators for determining the quality of silk. It can be seen from Table 2 that in 6 minutes, at P50 microwave power, the elongation and tensile strength of the monofilament after the treatment with polyvinyl alcohol gradually increased and decreased after 6 minutes. The bundle properties of the monofilament bundles finished by polyvinyl alcohol are improved, and the overall cohesion force enhances the tensile strength. Excessive microwave time will degrade the filamentous protein and reduce its strength and elongation[10-12].

4. Conclusions
The silk monofilament bundle was impregnated with a suitable concentration (2%) of polyvinyl alcohol, and crosslinked and cured by microwave heating at a suitable power (P50). The finished silk samples were characterized by infrared spectroscopy and X-ray powder diffraction, scanning electron microscopy and optical microscopy. The structure and appearance changes of the silk monofilament bundles were investigated. It is found that the finished monofilament bundles have good aggregation and comfortable feel and the chromaticity does not drop significantly. The tensile strength is slightly enhanced and the anti-friction ability is significantly enhanced. On this basis, further optimize the process conditions to lay the foundation for the anti-pilling finishing of corporate embroidery.

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