Structure and Properties of Partially Dissolved Silk Fibroin Fibers Modified with Rare Earth Chloride
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Key Words: rare earth treatment; fiber morphology; structure.

In this paper, we studied the morphology, aggregation structure and mechanical properties of partially dissolved silk fibers modified with lanthanum trichloride (LaCl₃) to find a new approach to improve natural shortcomings of silk fibers, including poor shrink resistance.

MATERIALS AND METHODS
1. Method of partial dissolution
Degummed mulberry silk of 47dtex (40/44D) x8 was used for the experiments. The ternary solution of CaCl₂, CH₃CH₂OH and H₂O was prepared in line with the mole ratio 1:2:8 to carry out partial dissolution, the degummed silk fiber was set in the calcium salt ternary solution with a (bath) rate of 1:100 at a temperature of 45°C for 1 hour.

2. Treatment techniques with rare earth
The partially dissolved silk fibers were treated in the rare earth solution of concentrations 0.1%, 0.3%, 0.5%, 0.7%, 1% at a rate of 1:100 and a temperature 40~45°C for 2 hours per treatment.

3. Test Methods
Several properties of these silk fibers were investigated using the following instrument; S-570 scanning electron microscope for microstructure test; YG020 electronic single silk strength machine for Single yarn extension strength test.

RESULTS AND DISCUSSION
1. Weight growth rate analysis

<table>
<thead>
<tr>
<th>Solution</th>
<th>0.1%</th>
<th>0.3%</th>
<th>0.5%</th>
<th>0.7%</th>
<th>1%</th>
</tr>
</thead>
<tbody>
<tr>
<td>RE treatment</td>
<td>+0.59%</td>
<td>+0.64%</td>
<td>+0.75%</td>
<td>+0.95%</td>
<td>+1.21%</td>
</tr>
<tr>
<td>PD RE treatment</td>
<td>+1.49%</td>
<td>+1.45%</td>
<td>+1.58%</td>
<td>+1.72%</td>
<td>+1.86%</td>
</tr>
</tbody>
</table>

The table shows the weight growth value of SF and PD SF fibers by immersion in different concentration percentage (from 0.1% to 1.0%) of RE solution. with the increase of the concentration of rare earth solution, both of the SF weight increase shows a tendency toward linear growth.

2. Fiber Morphology

Figure 1 show the surface morphology of SF fiber, and the PD SF fibers before and after RE treatment.
From Figure 1(a), the surface of untreated SF appeared highly smooth, showing only very fine longitudinal striation attributable to the fibrillar structure of the fiber as degummed SF fiber. When comparing Figure 1 (a, b), some shallow vertical lines on the surface of the PD SF fiber are evident. Figure 1 (b, c) shows that there appear to be variable amounts of foreign material deposits on the surface of the silk fibers after RE treatment.

![Fig 2 (a) (b) (c)](image)

Figure 2 show the cross-section morphology of untreated SF, and PD SF fibers before and after treatment with rare earth. From Figure 2(a), the surface of degummed untreated SF fiber is comparatively smooth. When degummed silk partially dissolved, many micro-pores appear inside fibers, and the extent of the cross-section increases and broken rims can be determined [Figure 2(b)]. But it cannot be determined that the PD SF fibers treated with RE are made looser and bear more pores [Figure 2 (b, c)].

3. WAXD Profiles

![Graph](image)

The figure is X-ray extension analysis for comparison of degummed SF fiber and PD SF fibers before and after RE treatment. In the Figure 5 for diffraction maximum 1, the 2θ diffraction peaks remain at 9.38°, 9.36°, 9.42°, while for diffraction maximum 2, the 2θ diffraction peaks remain at 20.64°, 20.74°, 20.54°. In the graph the forms of the three curves are similar and the peaks 2θ are almost the same, which means that RE treatment will not lead to radical changes the microstructure of the silk fibers. Among the three curves, the peaks strength of the PD SF fiber is the highest while the strength of the PD SF fiber after RE treatment is the lowest.