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# Fading of Silk Fabric Dyed with Phellodendron

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**Abstract.** Commercial silk habotai was dyed with phellodendron according to Chinese traditional dyeing method. After dyeing, the samples were quickly aged under ultraviolet lights in order to simulate the light fastness ageing of silk cultural relics during exhibition in museum. The results showed that discontinuous aging and continuous ageing resulted in different effects on the samples. High Performance Liquid Chromatography (HPLC) was used to analyze the changes happened under different aging treatments and to explain the reason of the poor light fastness of the silk fabrics dyed with phellodendron.

## Introduction

Many precious silk cultural relics tend to suffering from light aging affected by light sources of museum during exhibition. The degradation of silk fabric includes the degradation of silk fiber and the fading of its dyes. In general, the fading of dyes even happened before the degradation of the silk fiber itself. That is to say, dyes prevent the silk fiber from further degradation to certain extent. Therefore, it is very important to study the fading of dyes on silk fabrics.

Phellodendron was one of the most commonly used yellow dyes in ancient times. Silk dyed with it showed very pretty bright yellow color. However, silk cultural relics dyed by phellodendron tended to fading under exposure to the light sources in museum, due to poor light fastness of phellodendron, which calls for more care in exhibition. Light aging tests to simulate the degradation process which could happen in exhibition were designed and performed in this study, hoping to provide favorable evidences to protect silk cultural relics dyed with phellodendron.

## 1 Experimental

### 1.1 Materials

Silk habotai was obtained from Zhejiang Misai Silk Limited Corporation. Phellodendron was collected from Hunan province. Distilled water was used in all the processes.

### 1.2 Dyeing [1, 2]

50 g phellodendron was soaked into water for 24 hours, and then rinsed. Pigments were extracted from it in boiling water for three to four times, with half an hour treatment each time. Then all the extracted pigment solutions were poured into a stainless steel pot as a dye bath. Meanwhile, silk fabrics were rinsed, air dried and then put into the dye bath for dying at 60 °C for 40 minutes. The dyed samples were rinsed and further dyed by alum mordant for 10 minutes. After that, the samples were rinsed again and air dried for further treatment.

### 1.3 Accelerated ageing with UV light

Aging experiment was designed according to the standard ASTM G 154-06 as the following described [3].

**Continuous ageing**

20 pieces of the dyed samples were placed on the sample stage in order, with a 30 cm distance between the sample stage and the UV lights. The position of the samples was changed every one hour to ensure the uniformity of illumination. One sample was taken out at every 5 hours, and put into a box away from light. After 100 hours, all samples were taken out for tests.

**Discontinuous ageing**

20 samples were exposed under UV lights for five hours from the start. After exposure, one sample was taken out and put into a box away from light, while the others were put into another box away from light for 19 hours, and then exposed under UV lights again for 5 hours next day. In each following day, one sample was taken out after 5 hours illumination and protected from light illumination, while the others were kept away from light for 19 hour and then under exposure of UV lights for another 5 hours again on next day. After all samples were taken out, they were ready for tests.

### 1.4 Color difference measurement

Color difference was calculated using (CIE 1976) L*a*b* color difference formula as shown in equation (1).

$$\Delta E_{CIELAB} = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (1)$$

### 1.5 HPLC analysis

Chromatographic measurements were carried out using a Shimadzu HPLC system. The dyed samples were put into 400 ul solution with water/methanol/hydrochloric acid of the proportion of 1/1/2 (v/v/v). The mixture was heated in open Pyrex tubes on a heating block for 10 min at 105 $^{o}$C. After cooling, it was filtered through a porous polyethylene frit. The clean filtrate was dried in a vacuum desiccator. The dried residues were then mixed with methanol/water solution with the proportion of 1/1 (v/v) and injected into the HPLC instrument for testing [4].

## 2 Results and Discussion

### 2.1 Color difference

Figs. 1 and 2 show the color difference of continuous and discontinuous aged fabrics dyed with phellodendron. Clearly, the light fastness of phellodendron was quite poor. The fading of phellodendron happened at a very high rate within 20 hours' exposure and then followed by a lower rate after 20 hours' exposure. In addition, the fading rate of discontinuous ageing was lower compared with that of continuous ageing. The poor fastness of phellodendron could be explained as follows.

Berberine, a kind of cationic dye, is the main ingredient of phellodendron. When fibers were dyed with phellodendron, berberine molecules reacted with the anionic groups on the surface of silk fiber, such as carboxyl and hydroxyl groups. However, this reaction was quite limited. Once the reaction sites on the surface of silk fiber were used up, berberine molecules could not enter the fiber under the influence of space resistance. Therefore, the dyes existed on the surface of the fiber as a single molecular layer adsorption, which conformed to the Langmuir adsorption and unstable [5]. Moreover, due to the charge repulsion happened between the berberine cations, phellodendron dyes dispersed on the fiber surface just as a single molecular layer, which exposed large surface area and sites available to the reactions with oxygen, moisture and light, on the other side. Thus the fading rate was quite high from the start. Once the reaction of berberine cations with oxygen, moisture and light was ended, after 20 hours' exposure in this case, the fading rate tended to be lower [6]. In

addition, the cationic dye of phellodendron is very sensitive because of conjugated effect. It is much easier to be activated by light, and then transferred to the whole color system, leading to the fade and damage of fabrics. Therefore, the light fastness of fabric dyed with phellodendron is extremely poor. This change can also be seen from the SEM images shown in Fig. 3. The dyes on the surface of the silk fibers were damaged badly and almost disappeared after 100 h aging.

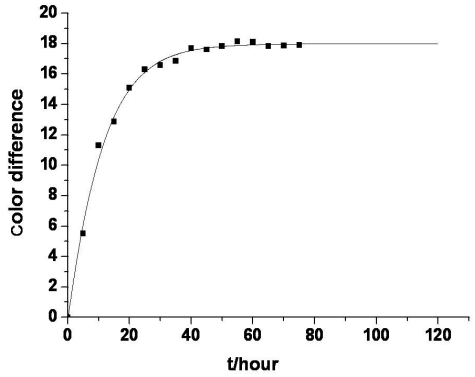



Fig .1 Color difference of continuous-aged fabric dyed by phellodendron

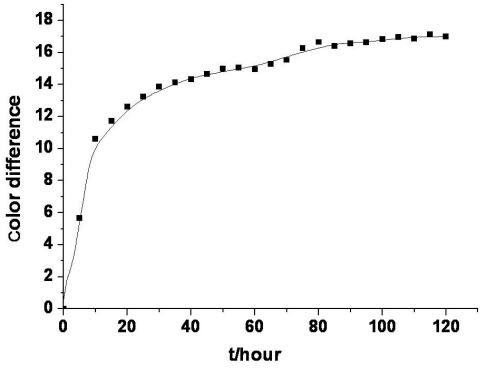
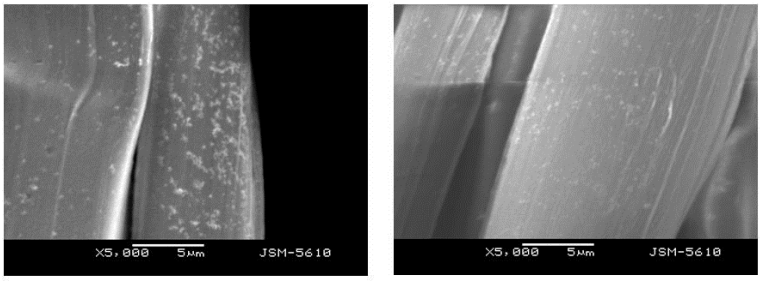



Fig. 2 Color difference of discontinuous-aged fabric dyed by phellodendron




(a) (b)

Fig. 3 SEM images of the fabrics dyed by phellodendron (a) before and (b) after 100h ageing

## 2.2 HPLC analysis

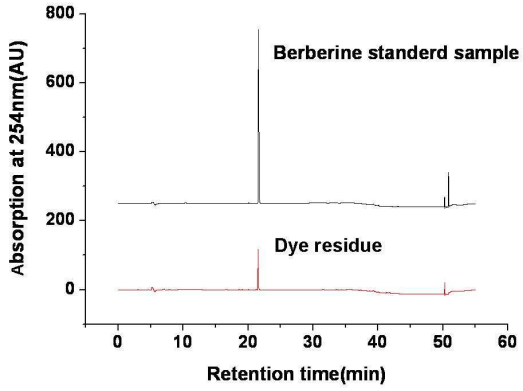



Fig. 4 HPLC chromatogram of the berberine standard sample and the dye residue

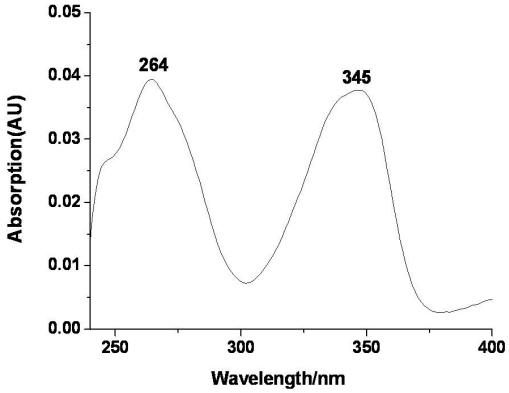



Fig. 5 UV spectrum of the dye residue from the fabric dyed with phellodendron

Fig. 4 shows the HPLC results of the dye residue from the fabric dyed with phellodendron and the standard sampled of berberine. The peak at the retention time of 21.5 minute showed the main ingredient of phellodendron-berberine, which was correspondent with the standard sample of berberine. Fig. 5 shows the UV spectrum of the dye residue from the fabric dyed by phellodendron. Two peaks are presented at 264 nm and 345 nm, which are specific peaks of berberine. So it is certain that the main ingredient of phellodendron is berberine.

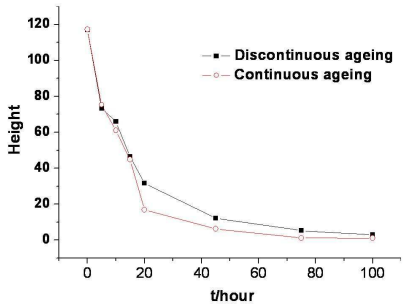



Fig. 6 HPLC peak height of the dye residues from the fabric dyed by phellodendron

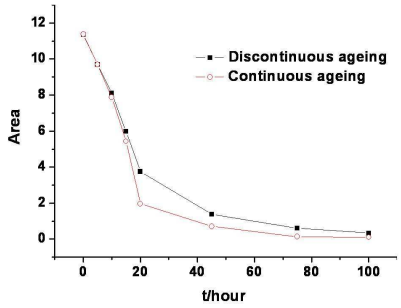



Fig. 7 HPLC peak area of the dye residues from the fabric dyed by phellodendron

Figs. 6 and 7 show the peak strength and adsorption area of the dye residues from the fabric dyed by phellodendron, relatively. These two figures actually show the change of berberine at different aging time. It can be seen that the height and area of the peaks drop quickly at the first 20 hours exposure and then drop slower later in both aging processes, with those of discontinuous aging presenting slower tendency. After about 100 hours exposure, the height and area of the peaks were close to zero, which meant berberine was almost exhausted. The tendencies corresponded with those of color difference tests shown in Figs. 1 and 2. Therefore, if it is difficult to measure the color difference once silk fabric was in serious damaged conditions or the surface of the silk fabric was not smooth, we can judge the light fastness of silk fabric by the results of HPLC test [7, 8]. Moreover, the HPLC measurement can be performed with little sampling and has high sensitivity.

## 3 Conclusions

Silk habutai was dyed by phellodendron and its light fastness was studied. The results indicated that discontinuous accelerated aging showed lower damaging effect on fading than that of continuous accelerated aging. Phellodendron dyed silk cultural relics should be kept off light regularly in exhibition. Phellodendron dyed fabrics were easily faded because of the unstable nature of berberine as the main ingredient in phellodendron. HPLC test can be applied to measure light fastness if color fastness test is not possible.

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