

# The colour and micro-structure change of the aged madder-dyed silk fabric adding with consolidation material of EGDE/Ala

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**Abstract.** A consolidated method by EGDE (ethylene glycol diglycidyl ether) and Ala (alanine) was used for fragile ancient silk fabrics protection. Therefore, an investigation has been carried out to figure out the effect of consolidation material in the system. Silk fibers were dyed with madder and then exposed to UV light beams for accelerated aging. The aged madder-dyed samples were treated with EGDE aqueous solution by spraying, and then treated with Ala aqueous solution by the same way on 10 minutes later. The samples were investigated by colour measurement and scanning electron microscopy (SEM). It has been observed that there is an obvious change of colour of samples after consolidation material treatment. The surface of the aged madder-dyed silk fabric were covered with EGDE and Ala thus protecting the silk fibers.

## 1. Introduction

Historic silk textiles as Chinese cultural heritages are very precious and of great value in research. For the ancient silk fabrics were deteriorated by water, light, heat and microorganisms [1-4], it is very important to protect historical silk fabric relics for further displays and researches. Many consolidation materials and methods were adopted in the protection of ancient silk fabrics. Such as weave [5], mounting [6], polymers [7], resin, adhesive [8] and so on. But the applications are limited by the problems, like high fabric stiffness, color difference or yellowing of silk fabrics and lead to potential harm to silks in the long term and so on, produced in the protection processes.

EGDE as an epoxide with bifunctional groups has high reactivity with amines, alcohols, phenols, carboxylic acids and thiols [9]. Consequently, EGDE was widely used in the preparation of three-dimensional polymers [10] and gels [11] as a cross-linking agent. Moreover, EGDE has been also used to modify polyrotaxane fibers [12], silk fibers [13] and silk fabrics [9, 14] to improve properties such as elongation at break, moisture regain, thermal stability and so on. In this study, EGDE was used as material to consolidate aged madder-dyed samples. Ala, which is homologous and compatible with silk, was used to consolidate fragile samples with the support of EGDE.

EGDE can be reacted with Ala and silk fabrics by simple spraying at low temperature, which is easy to perform and has significant effect on the improvements of mechanical properties compared with other materials [15, 16]. Therefore, EGDE was adopted as a material for treatment of very weak historic silk fabrics. Though we have some researches on properties of the aged silk fabric (undyed)



adding with consolidation material of EGDG, there is still no work carried out to research the effect of dyed silk fabric on properties especially in color difference.

The consolidation materials of EGDE and Ala were added into artificial aged madder-dyed silk fabric by spraying at room temperature, and the effect of the aged madder-dyed silk fabric adding with consolidation material of EGDG/Ala on color and micro-structure were studied.

## 2. Experimental

### 2.1. Materials

Plain weaved reference silk fabrics (Hangzhou Fusi Textile Co.,Ltd) was used for experiments. Madder was purchased from Chinese medicinal herbs store, China. EGDE used in this study was supplied by Nagase ChemteX Co. Ltd. (Japan) as consolidation materials. Alanine was purchased from Aladdin Chemistry Co., Ltd. Distilled water was used in the experiments.

### 2.2. Preparation of control and treated samples

The madder were soaked in the water for 24 h. Then the water which the madder were soaked in was heated to boiling temperature for 90 min. Water was added during the heating process until the extract was cooled and then filtered two times to get the solution of Dye. The silk fabric was treated in water at 100°C for 30 min, then rinsed and air dried. The dyeing procedure was performed in a beaker with the liquor ratio of 1:50 (For 1 g of silk fabric a dye solution of 50 mL is applied). 4g of silk fabric were used in this study. The silk fabrics were treated with the dye solution and then it was brought slowly to the 80°C for 60 min. Then the samples were removed, squeezed, and air dried. After dyeing, the dyed samples were rinsed with hot water at 80°C and then washed with hot water at 40°C. The unfixed dyestuff was removed by washing twice with cold water, and the dyed silk fabric were removed, squeezed, and air dried. The dyed samples were cut up into small pieces (2 × 10 cm). The small piece of dyed samples were put in UV Accelerated Weathering tester which equipped with 500 watt UV lamp, 70°C, 40% relative humidity (RH) for 30 days. Then the artificial aged madder-dyed silk fabric samples (control samples) were prepared. The aged madder-dyed silk samples were smoothly put on a net frame. The aged madder-dyed samples were treated with EGDE aqueous solution of 2.5 wt% by spraying, and Ala solution with the concentration of 1.5 wt% was gently sprayed on the samples which were treated with EGDE after 10 minutes. Then the samples were dried at 25°C for tests.

### 2.3. Characterization of the artificially aged madder-dyed silk fabric samples and the samples treated by consolidation materials

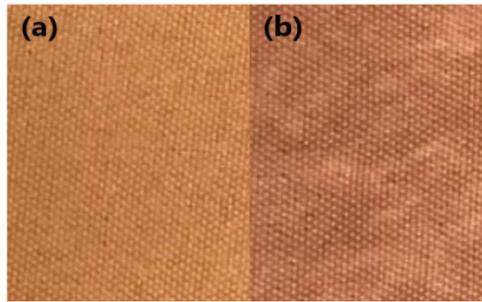
The Colour differences of samples were measured using a Konica Minolta spectrophotometer (CM-700D, Japan) interfaced with a personal computer. The colour characteristics ( $L^*a^*b^*$ ) of the aged madder-dyed silk fabric were assessed for all samples treated in the presence and absence of consolidation materials. Colour differences between the samples were determined by the following equation:

$$\Delta E_{ab}^* = \sqrt{[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]} \quad (1)$$

Samples were cut into 5\*5mm and mounted on aluminium stubs by using electrically conducting copper tape. Then the samples were sputtered with gold for 60 s at 15 mA. The morphology of the surface of the control samples in comparison to the two kinds of artificially aged samples were observed by using SEM (JSM-5610LV, Japan) under the voltage of 5kv and the working distance of 15mm .

## 3. Results and Discussion

### 3.1. Colour difference



**Figure 1.** Images of the aged madder-dyed silk fabrics of (a) control, (b) EGDE\Ala treated

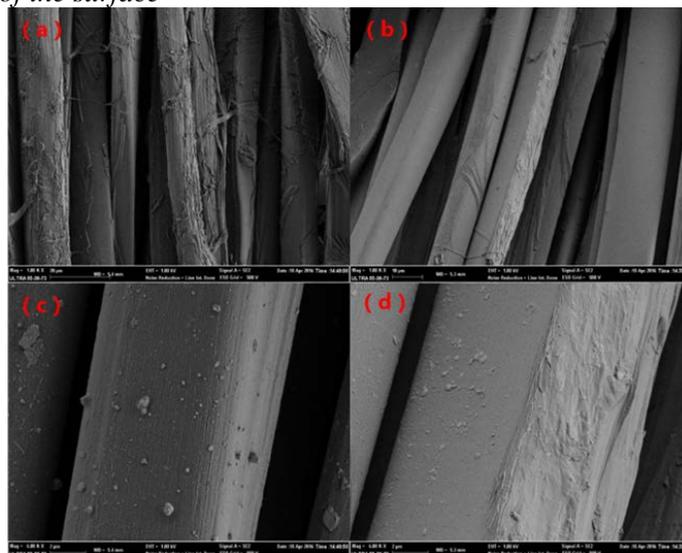
Figure 1 shows that the aged madder-dyed silk fabrics which treated with consolidation material appear in shades of red, and darker than the control sample. It has been observed that there is an obvious change of colour of samples after EGDE\Ala treatment.

**Table 1.** Colour difference of the aged madder-dyed silk fabrics and treated samples

	$\Delta L$	$\Delta a$	$\Delta b$	$\Delta E$
Colour difference	-5.38	2.14	-0.75	5.84

In order to exactly evaluate whether consolidation material of EGDE/Ala has an influence on colour, chromatic values of aged madder-dyed silk fabric before and after treatment were tested. The result in Table 1 shows that the total colour difference ( $\Delta E$ ) between control sample and treated sample is about 5.84. The value more than 1.0 suggests that the treatment is not acceptable in the protection of ancient silk fabrics [17]. The CIELAB system also provides a psychometric index of lightness  $L^*$  which is about -5.38. This negative values indicated that the colour of treated sample distinctly darker than the untreated one. One colour coordinates is  $a^*$  which is about 2.14 (reddish coloration), and the other is  $b^*$  which is about -0.75 (bluish coloration). The greater of the absolute value shows the more obvious of the colour difference.

### 3.2. The morphology of the surface



**Figure 2.** SEM images of the aged madder-dyed silk fabrics ((a) and (c)) and EGDE\Ala treated samples ((b) and (d))

The samples were subjected to SEM analysis to evaluate morphological changes that may have occurred before and after EGDE\Ala treatment, which would led to changes in physical and mechanical properties. Scanning electron microscopy (SEM) images of fragile silk fabrics before and after treatment are shown in Fig.2. As shown in Fig.2 (a), the surface of the aged madder-dyed silk fabrics are rough and destroyed. The surface morphology of silk fiber wasn't changed significantly, as depicted in Fig.2 (b). These results suggest that treated with consolidation material of EGDG and Ala has obvious influence on its appearance with smooth the surface. EGDE and Ala might react with silk fabrics, and form a layer of film on the surface of silk fabrics (fig.2(c) and (d)), thus increasing the breaking strength and protect the silk fibers.

#### 4. Conclusions

The aims of this paper is to study the effect of the aged madder-dyed silk fabric adding with consolidation material of EGDG/Ala on colour and micro-structure. The results concluded that the addition of consolidation material of EGDG/Ala affected the colour and micro-structure of the aged madder-dyed silk fabric. It has been obtained that there is an obvious change of colour of samples after EGDE\Ala treatment. EGDE and Ala were covered in the surface of silk fabrics to protect the silk fibers. The colour and micro-structure change probably results from the triadic interactions of EGDE, Ala and silk fabric. However, the detailed mechanism and property is unclear. Therefore, we need to do further research based on this work.

#### Acknowledgements

We thank to financial support from National Key Technology R&D Program (2013BAH58F01-02) and Special Funds from the Administration of Cultural Heritage of Zhejiang Province (2016011).

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