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Key to the conservation of calligraphy and painting relics in collection: proposing a lighting damage evaluation method

Zhihui Wei¹, Zhichao Feng¹ and Huijiao Tan^{2*}

Abstract

As an important supporting material for calligraphy and painting relics in collections, the silk substrate is generally in acidification. And owing to an extremely high light response, it is very prone to different forms of lighting damage, including color and mechanical aspects. Considering that the Light Emitting Diode (LED) with flexible spectral compositions is widely used in the lighting environment of collections, the spectral responsivity of acidified silk substrates is the core issue concerning the long-term conservation of calligraphy and painting relics. In this study, based on the accelerated aging experiment of nine narrowband LEDs with different wavelengths (λ) in the visible range on silk samples with different acidification degrees (pH values), the infrared spectrum was measured non periodically. Processed with the Principal Component Analysis (PCA), the obtained ΔPC_1 was used to evaluate the lighting damage of silk samples. Based on the damage change curve, its spectral responsivity function, that is, the mathematical relationship between lighting damage and λ and pH values, was further fitted. Combined with the previous research on the spectral responsivity of colorants, an evaluation method for lighting damage was proposed, which is the key to the conservation of calligraphy and painting relics.

Keywords Calligraphy and painting relics, Acidified silk substrates, Lighting damage, Spectral responsivity, FTIR, PCA

Introduction

Calligraphy and painting relics (hereinafter referred to as relics) in collections, known as a huge stock and rich content, are an important part of the world cultural heritage and have important historical and artistic values. With the popularization of the concept of “relics first, conservation first” [1], it is particularly important to carry out conservation for the relics [2]. That is to say, identifying and evaluating environmental factors that may cause damage to the relics provides a theoretical basis

for developing a series of conservation methods [3–5]. Lighting in the museum, as an environmental factor for the preservation and display of the relics, is an important reason for the damage to them [2, 6]. This is because their two components, the colorant covering substrate and the blank substrate, are defined as high light responsive materials [7]. Especially, the silk substrate that first appeared during the Warring States period (~256 B. C.) is composed of the high polymer of proteins, which are prone to different forms of lighting damage, including the color aspect like yellowing, and the mechanical aspect like deformation and embrittlement. Therefore, lighting damage evaluation is the key to the conservation of the relics.

It is mentioned in the technical report of Commission Internationale de l'Eclairage (CIE) that the lighting damage is a function of exposure and spectral responsivity [7]. For materials with high light responsivity such

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as colorants and silk substrates, there are recommended values of exposure in different lighting standards [7–9]. However, the importance of research on the spectral responsivity is highlighted by the application of Light Emitting Diodes (LEDs), for their flexible spectral compositions bring great challenges to the conservation of the relics.

To our knowledge, most of the related studies tend to focus on colorants by considering their interaction with substrates [10–12], with little consideration given to the blank substrate. Liu et al. studied the spectral responsivity of historical paper under irradiation of three narrowband lights by measuring reflection spectra and color difference [13]. Through the accelerated light aging experiment on silk substrates, Dang et al. obtained its spectral responsivity function based on the color difference [14]. It can be seen that the color difference is a commonly used method to evaluate the lighting damage, whereas it is only applicable to the color damage of silk substrates. Halvorson et al. evaluated changes in mechanical properties such as tensile strength of silk based on tensile tests [15]. If combined with methods for evaluating color and mechanical damage, the different damage rules obtained could bring great inconvenience and limitation to the practical application. For the silk substrate, the lighting damage on the macro level is due to changes in molecular structures caused by the absorption of photon energy [12]. Considering the advantages of non-destructive measurement without sample preparation, spectroscopy, as a means to analyze the macro properties of materials from molecular structures, is particularly suitable for evaluating the lighting damage to silk substrates [6]. Dang et al. obtained the lighting damage coefficients of four narrowband lights on paper and silk substrates by analyzing the changes in characteristic Raman peak intensity [16]. Due to the fluorescence effect of Raman spectroscopy in testing organic materials, infrared spectroscopy is a more commonly used method for studying proteins in silk substrates [17, 18]. Kopersk proposed several damage evaluation indicators based on infrared spectral peaks to assess the damage to silk under water vapor and oxygen from aspects of crystallinity, degrees of oxidation and polymerization [19]. However, in the existing studies, evaluating changes in one [20] or multiple molecular structures [19] based on calculating the infrared spectral peak intensity ratio may, on the one hand, omit unexpected but important phenomena caused by changes in other molecular structures during the damage process, and on the other hand, not be conducive to practice owing to different damage rules based on changes in multiple molecular structures.

Chemometrics is a discipline that links the measured value of a chemical system with its state through

statistical or mathematical methods, that is, to extract information to the maximum extent to explain the changes in the system. It is a feasible method to extract information from the infrared spectral dataset with chemometrics [21, 22]. Principal Component Analysis (PCA) is a classical method for information extraction in data [23]. For a given infrared spectral datasets (in the form of matrix X , where each row and column represent a spectrum and wavenumber respectively), they can be projected by PCA as points in the principal component space. That is to say, the principal components are represented as linear combinations to characterize the original high-dimensional infrared spectral dataset [24], as shown in Eq. (1).

$$X = PC_1 \cdot t_1 + PC_2 \cdot t_2 + \dots + PC_n \cdot t_n \quad (1)$$

where PC and t are the two vectors of the principal component, respectively. The score vector PC contains the score values of each spectrum, which is used to illustrate how the spectrum is related to other spectra. The load vector t reveals which spectral change characteristics in the original spectrum captured by the specific PC [25]. On the premise of the principal component achieving at least 85% interpretation of the infrared spectral dataset, its change in the score value can be used to quantify the changes in the dataset, i.e., the lighting damage to samples [26]. In addition, the load vector t captures the spectral change characteristics of samples, which explains changes in molecular structures during the process of lighting damage.

Apart from the issue concerning methods, the above study seems to ignore a common material property of silk substrates, namely acidification [27, 28]. An important source of acidification is the additives retained in the production process of silk substrates, namely, the mixture of animal glue and alum ($KAl(SO_4)_2 \cdot 12H_2O$) [29]. The hydrolysis of Al^{3+} could promote the acidification of silk substrates, seriously affecting its durability. Kim et al. pointed out that the increase of acidity is directly related to the damage to silk [30]. As far as we know, there is little study on spectral responsivity of acidified silk substrates. However, as Pastorelli et al. said, there is an interaction between the influence of environment and material properties on itself [31]. Therefore, we believe that it is urgent to study the spectral responsivity of acidified silk substrates with the spectral quantification method of infrared spectroscopy combined with PCA.

In this study, based on the accelerated aging experiment of nine narrowband LEDs in the visible range on silk samples with different acidification degrees, the spectral quantification method was proposed to study their spectral responsivity. In combination with the previous study on the spectral responsivity of colorants, an

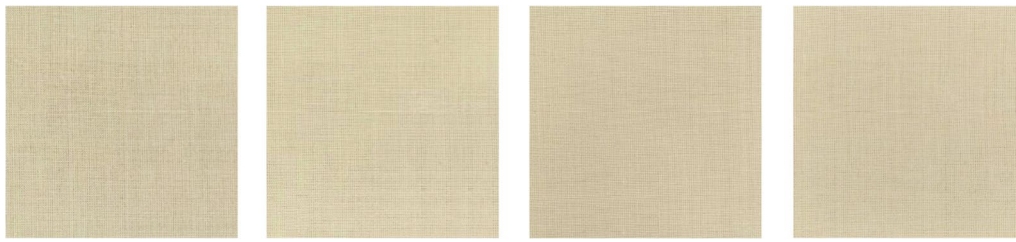


Fig. 1 Silk samples with pH value increasing from left to right

Table 1 Proportions of animal glue and alum for silk samples with different pH values

Samples	Proportions(g)			pH values
	Animal glue	Alum	Deionized water	
Silk	0.5	2.3	100.0	3.5
	0.5	0.2	100.0	4.0
	0.5	0.1	100.0	5.0
	0.0	0.0	0	6.0

evaluation method for lighting damage to the relics was proposed.

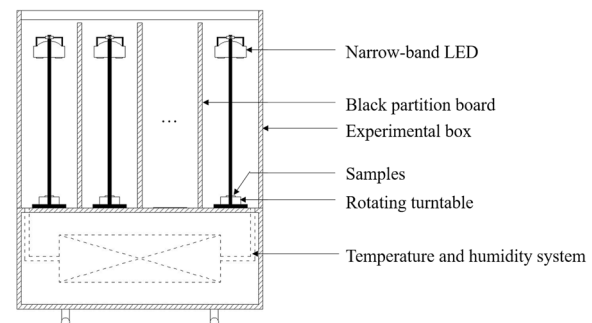
Materials and methods

Samples

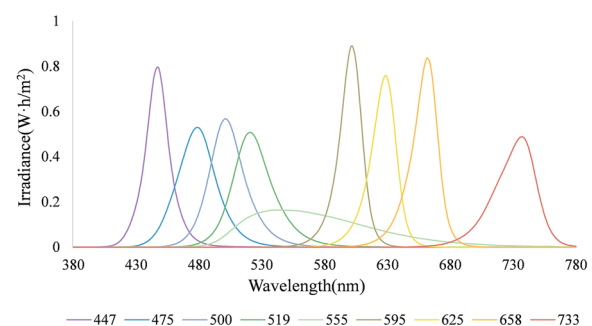
The National Museum of China has kindly provided us with several silk substrates as experimental samples. Considering the acidification sources, the solution made of different proportions of animal glue and alum was brushed on silk samples to acidify them. Using cold extraction method [32], the pH values of silk samples were determined as 3.5, 4.0, 5.0, and 6.0 respectively, basically covering the pH range involved in the literature [30]. The silk sample is shown in Fig. 1, whose specific information is shown in Table 1.

Accelerated aging experiment

Accelerated aging experiment was carried out in the experimental box with automatic temperature and humidity adjustment function in the all-dark optical laboratory, as shown in Fig. 2a. In the experimental box divided into nine independent spaces with black partition boards, nine narrowband LEDs within the visible light range were respectively installed on the top, vertically irradiating downward silk samples at an interval of 54.5 cm. Its spectral power distributions (SPDs) were measured by the spectroradiometer (Photo Research PR-670), as shown in Fig. 2b. In order to improve the irradiation uniformity, samples in nine independent spaces were all placed on an automatic rotary table with



(a)



(b)

Fig. 2 a Experimental setup. b SPDs of nine narrowband LEDs

a speed of 0.5 r/min, controlling the irradiance on sample surface about 20 W/m². During the experiment, lighting evaluation indicator was obtained through a synchronous analysis of the infrared spectral data by PCA. On that basis, when the variation of lighting damage with exposure reaches a stable stage, the experiment was ended, with a cumulative exposure of 984 h and a total exposure of 19,680 W·h/m².

Data measurement

Before and after the accelerated aging experiment, the infrared spectra of silk samples were measured non periodically. Considering that the damage rate tends to change from fast to slow [7], intensive measurements were carried out in the early stage of the experiment. A

total of 19 measurements was taken plus the one before irradiation.

The infrared spectrum was measured using FTIR Spectrometer (Bruker INVENIO-R), which is equipped with DLATGS detector, an accessory of Attenuated Total Reflectance (ATR), and diamond crystal. The spectrum was obtained in absorbance, recorded from 400 to 4000 cm^{-1} , with 4 cm^{-1} resolution and 32 scans. The atmospheric background at the time was collected and deducted for each spectrum. By marking the ATR accessory platform and using its relative position with samples, the repeatability of each measurement was ensured as far as possible.

Taking the silk sample with pH 5.0 as an example, a detailed explanation of its infrared spectrum before irradiation is provided, as shown in Fig. 3. The silk substrate is composed of proteins, containing 18 amino acids, mainly glycine (Gly), alanine (Ala), serine (Ser), tyrosine (Tyr), etc. The two peptide chains of -Gly-Gly- and -Gly-Ala- form the main sequence of amino acids, with infrared spectral peaks at 1001 cm^{-1} and 976 cm^{-1} , respectively [33]. 1160 cm^{-1} is attributed to the vibration of tyrosine phenolic hydroxyl groups [34]. These amino acids form a specific secondary structure of silk proteins, with infrared spectral peaks at 1230–1265 cm^{-1} (amide III, mainly C=O and C–N stretching vibration), 1515–1520 cm^{-1} (amide II, mainly N–H bending vibration), and 1600–1690 cm^{-1} (amide I, mainly C=O and C–N stretching vibration) [35–37]. In addition, there is infrared spectral peak attributed to N–H stretching vibration at 3290–3300 cm^{-1} [35]. Therefore, the infrared spectral peaks within the aforementioned wavenumber range are of concern for the analysis of lighting damage to silk samples.

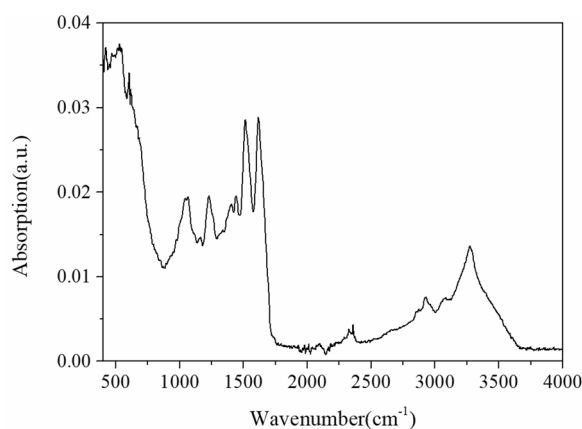


Fig. 3 Infrared spectrum of the silk sample with pH 5.0

Data analysis

The measured data of each silk sample are presented as a high-dimensional dataset of infrared spectra, which was analyzed by PCA. Before the calculation, the infrared spectral dataset of each sample was averaged and centralized, which ensures them explained by changes around the mean without changing their statistical structure [38, 39]. After the calculation using SIMCA 14.1 (Umetrics, Sweden), the corresponding principal components were obtained for each sample's dataset. There is the highest interpretation variance in PC_1 and PC_2 , whose score values can be used to obtain the score plot in order to observe the distribution trend of silk samples before and after the irradiation. The loading plot can be used to explain the reason for the change in the score vector, i.e., the change in the infrared spectra characterizing lighting damage.

Taking the silk sample with pH 5.0 under the irradiation of 595 nm narrowband LED as an example, the score and loading plots are explained in Fig. 4, which is similar to those under other narrowband LEDs. According to Fig. 4a, the points projected onto the principal component space by the samples pre-irradiation and post-irradiation are well separated along the PC_1 -axis, which means that there is lighting damage during the accelerated aging experiment. Meanwhile, the interpretation variance of PC_1 has exceeds 90%. Thus, changes in the score value (ΔPC_1) can be used to characterize the lighting damage to silk samples. In the loading plot in Fig. 4b, the wavenumber range with higher loading weights can be used to explain the infrared spectral changes captured by PC_1 , i.e., changes in the molecular structure that cause lighting damage to silk samples. Within this wavenumber range, ~ 1000 cm^{-1} , 1160 cm^{-1} , 1230–1265 cm^{-1} , and 3290–3300 cm^{-1} belongs to the -Gly-Gly- and -Gly-Ala- peptide chains, tyrosine, amide III, and N–H stretching vibrations, respectively. Among them, the infrared spectral peak intensity ratio of I_{1264}/I_{1231} in the amide III is usually used for calculating crystallinity [33]. Research has shown that the crystalline region of silk protein is surrounded by some disordered peptide chains, forming an amorphous region [40]. Considering that the crystalline and amorphous regions respectively endow silk with strength and elasticity, crystallinity is considered an important factor related to the mechanical properties of silk [41], whose change usually results from the weakening or destruction of hydrogen bonding between peptide chains [37]. In addition, the color change of silk is closely related to the photodegradation of tyrosine [42]. Therefore, the spectral change information extracted from the infrared spectral dataset by PCA can scientifically reflect the change in molecular

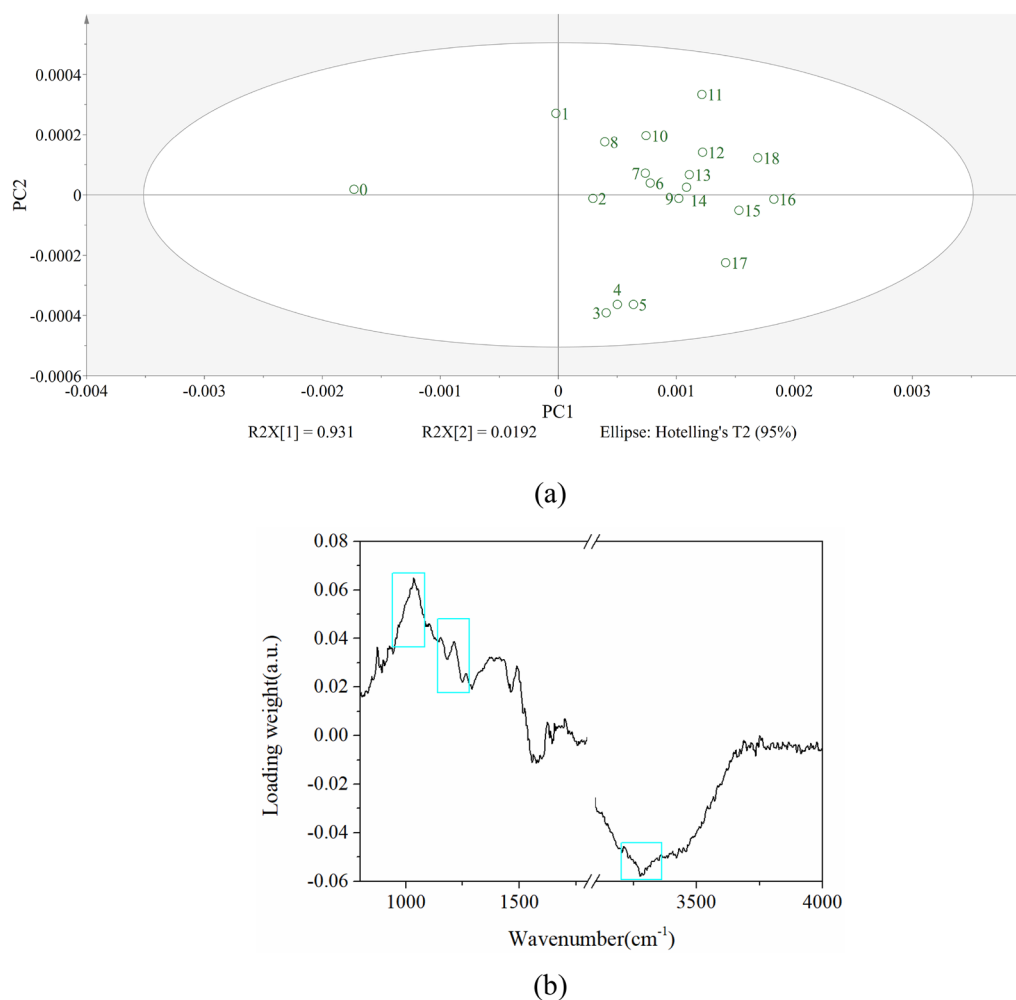


Fig. 4 **a** Score plot and **b** Loading plot of pH 5.0 silk sample under the irradiation of 595 nm narrowband LED (numbers 0 and 1–18 represent 19 tests before and after irradiation, respectively)

structures that causes the lighting damage to silk samples, thus verifying the reliability of this method.

Results and discussion

Variation of spectral responsivity of acidified silk substrates

Based on PCA calculation, changes in the score value (ΔPC_1) were used to characterize the variation of lighting damage with exposure. As the exposure increases, the lighting damage to silk samples increase, with the rate decreasing. Thus, using the Eq. (2) of the power function, the mathematical models of lighting damage variation of silk samples with different acidification degrees with exposure were fitted, as shown in Fig. 5, whose goodness of fit R^2 is between 0.85–0.98.

$$D = a \cdot Q^b \quad (2)$$

where D represents lighting damage, a and b are constants, and Q is exposure.

As shown in Fig. 6, taking silk samples with pH 5.0 as an example, the curve of its damage rate and exposure was obtained through derivative. It can be found that the damage rate decreases rapidly with increasing exposure, and finally approaches zero. That is to say, there is a damage inflection point, where the lighting damage has tended to remain unchanged. When the damage rate of nine narrowband LEDs to silk samples approaches zero, that is, reaching the exposure corresponding to the damage inflection point, the spectral responsivity is basically stable. For the ancient relics, although its historical information before the excavation cannot be verified, it is generally believed that the silk substrate has experienced a certain degree of damage, resulting in a relatively slow damage rate at present. On the premise of meeting

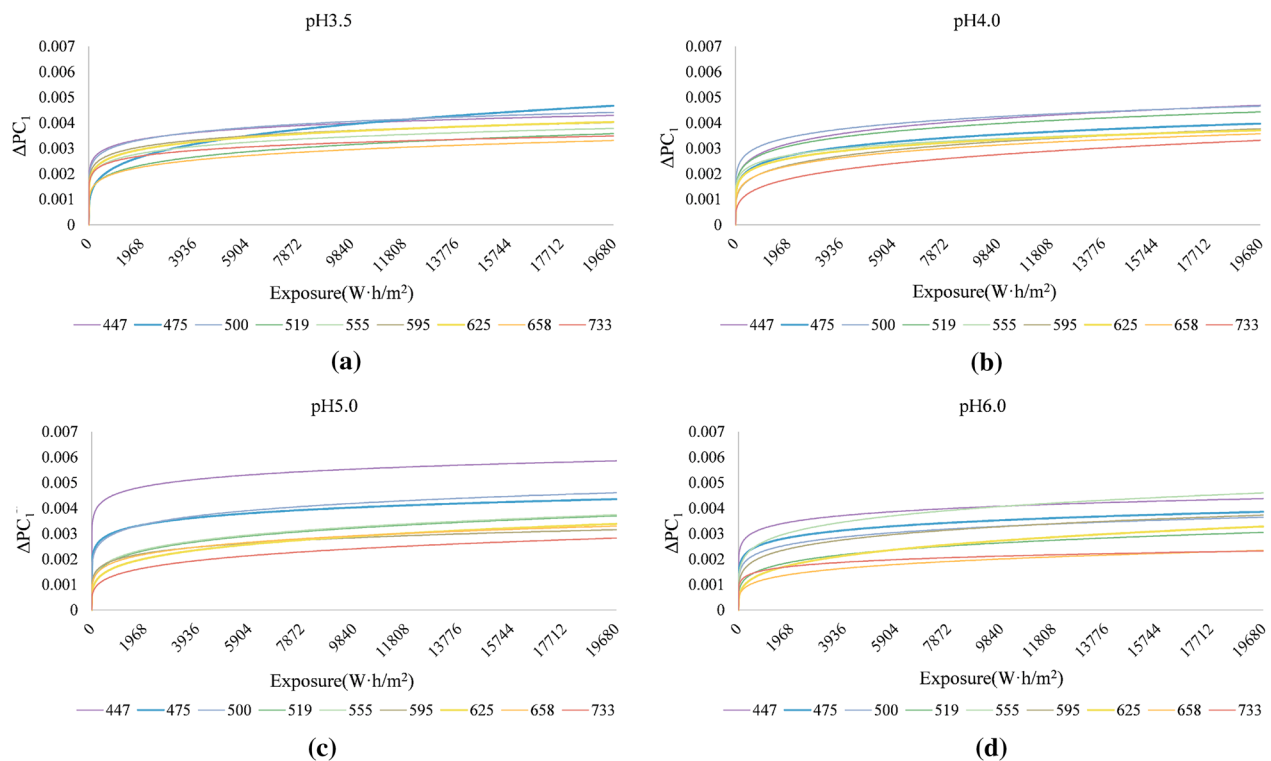


Fig. 5 Mathematical models of lighting damage variation of silk samples with **a** pH 3.5, **b** pH 4.0, **c** pH 5.0, and **d** pH 6.0 with exposure

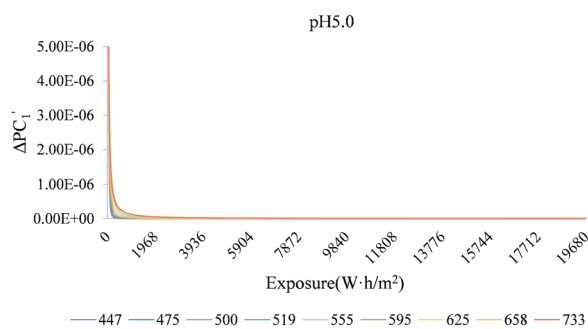


Fig. 6 Mathematical models of lighting damage rate variation of silk samples at pH 5.0 with exposure

the universality requirements brought about by the huge stock of relics, in order to make the results obtained in the accelerated aging experiment applicable to the silk substrate in relics, the lighting damage characterized by the corresponding exposure of the damage inflection point should be considered to further analyze its spectral responsivity. For the determination of the damage inflection point, we can refer to the derivation method proposed by Svensson et al. [26]. When the derivative of damage rate is less than 2×10^{-12} (noise level), it can be

Table 2 Exposure corresponding to the damage inflection point of silk samples with different acidification degrees

Acidification degrees	pH3.5	pH4.0	pH5.0	pH6.0
Exposure(W·h/m ²)	9298	10,283	6777	10,323

judged that the damage inflection point is reached. The exposure corresponding to the damage inflection point of silk samples with different acidification degrees is shown in Table 2.

The lighting damage coefficients of silk samples at the beginning, inflection point, and end of the experiment were calculated according to Fig. 5, which were normalized based on the coefficients of the 447 nm narrowband LED. The obtained relative lighting damage coefficients were used to plot the spectral response curves of silk samples with different degrees of acidification under three exposure levels, as shown in Fig. 7. It can be seen that, compared with the beginning exposure (20 W·h/m^2), the response curves of silk samples at the exposure corresponding to the damage inflection point has largely approached the curve at the end ($19,860 \text{ W·h/m}^2$). Thus, the spectral responsivity at the inflection point can be regarded as relatively stable.

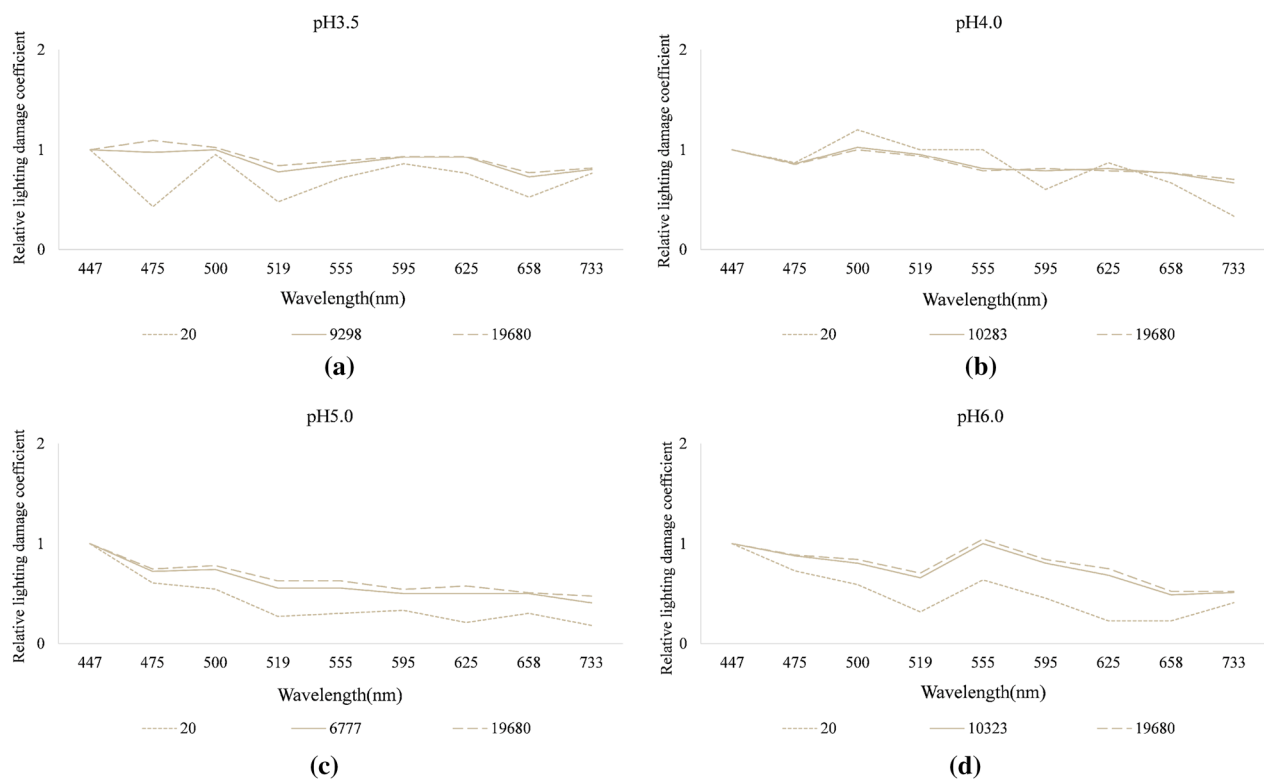


Fig. 7 Response curves of silk samples with **a** pH 3.5, **b** pH 4.0, **c** pH 5.0, and **d** pH 6.0 under the three exposure levels

Spectral responsivity function of acidified silk substrates and its application

Based on the relative lighting damage coefficient at the damage inflection point in Fig. 7, the spectral responsivity of acidified silk samples was further discussed, whose mathematical model was drawn as Fig. 8. It can be found

that the higher the λ and pH value is, the lower the lighting damage is. Accordingly, the spectral responsivity function of the silk samples was fitted as Eq. (3), whose goodness of fit R^2 is 0.8702. On that basis, the lighting damage to silk substrates with any degree of acidification can be calculated under the irradiation of light sources with any spectral composition.

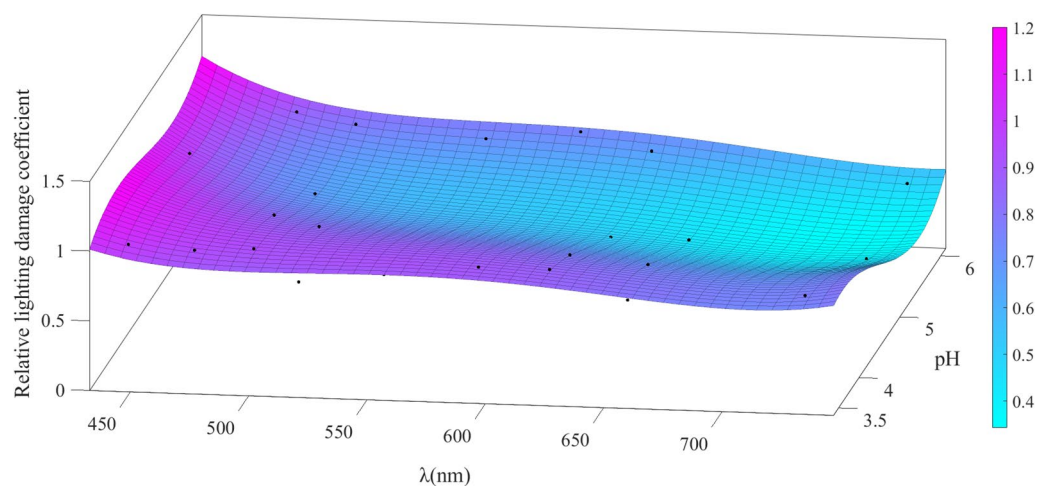


Fig. 8 Spectral responsivity mathematical model of silk samples with different acidification degrees

$$f(\lambda, pH) = 12.16 - 0.2481 \cdot \lambda + 19.97 \cdot pH + 0.0008413 \cdot \lambda^2 - 0.05381 \cdot \lambda \cdot pH - 2.217 \cdot pH^2 \\ - 1.106e - 06 \cdot \lambda^3 + 6.473e - 05 \cdot \lambda^2 \cdot pH + 0.002829 \cdot \lambda \cdot pH^2 + 0.07976 \cdot pH^3 + 4.965e - 10 \cdot \lambda^4 \\ - 1.964e - 08 \cdot \lambda^3 \cdot pH - 3.01e - 06 \cdot \lambda^2 \cdot pH^2 + 6.918e - 05 \cdot \lambda \cdot pH^3 \quad (3)$$

Considering the characteristics of the relics, it consists of two parts, blank substrates and colorants covering the substrate. Compared with the complex damage form in the former, the lighting damage of the latter is presented as a change in color, which is commonly evaluated by the color difference. In combination with the spectral responsivity function of colorants proposed by Dang et al. [10], it is feasible to propose an evaluation method for lighting damage to the relics. The specific process is as follows.

(1) Determining essential information of the relics.

First, the colorants and substrates in relics should be determined by characteristic peaks of Raman spectroscopy and infrared spectroscopy [43, 44]; Calculating spectral responsivity function of second, the respective weights W_c and W_s should be determined by counting the pixel numbers of both [45]; finally, the pH value of silk substrates should be measured.

(2) Calculating spectral responsivity function of relics.

First, the corresponding spectral responsivity function $D_c(\lambda)$ and $D_s(\lambda, pH)$ should be obtained respectively according to the composition of colorants and silk substrates in relics, substituting the measured pH value in the latter; second, the spectral responsivity function of relics should be calculated according to the Eq. (4).

$$f(\lambda, pH) = W_c \cdot f_c(\lambda) + W_s \cdot f_s(\lambda, pH) \quad (4)$$

(3) Calculating lighting damage to relics.

The technical report of CIE defines the lighting damage of relics as a function of exposure and spectral responsivity, only considering the lighting damage from the perspective of light sources [7]. However, this study verified that the degree of acidification, a material property, also has an impact on the spectral response of silk substrates, which further affects the relics.

First, the $S(\lambda)$ (i.e., SPD) of the light source should be measured with the PR-670 spectroradiometer; second, according to Eq. (5), the lighting damage D to relics can be calculated.

$$D = Q \cdot \int_{380}^{780} S(\lambda) \cdot f(\lambda, pH) d\lambda \quad (5)$$

Discussions

The results indicate that the spectral responsivity of silk substrates exhibits the rule that damage decreases with increasing wavelength. It is consistent with the research results of Dang [14] and Wang [46] on silk substrates in the visible light range, although they chose the color difference as the evaluation indicator. The results of this study indicate that the spectral responsivity of silk substrates with different degrees of acidification varies, which means that it is necessary to consider its own degree of acidification when calculating the lighting damage to silk substrates.

Relics, visitors, and staff, as the main elements of museums, have different concerns about the lighting environment, which brings it with the multi-objective lighting requirements. The current study and application trend is to prioritize reducing lighting damage while also improving their lighting quality and efficiency [2, 6]. Gargano proposed a new lighting method by reducing the spectral composition absorbed by materials in the light source, which not only reduces lighting damage but also improves lighting efficiency [47]. Lucchi proposed a simplified evaluation method that assesses the environmental and energy quality of museums, identifies potential risks, and determines conservation methods. Among them, lighting is an important cause of damage to the relics [5]. The lighting damage evaluation is the theoretical basis for carrying out conservation practices from the perspective of lighting. It achieves a reasonable selection of lighting sources by comparing the lighting damage caused by them on the relics, especially in the context of LEDs with flexible spectral compositions widely used in museums.

Durmus proposed a voxelated 3-D volume to balance the multi-objective lighting requirements, which quantifies the volume formed by indicators such as lighting damage, lighting quality, and lighting efficiency to compare the performance of different light sources. This method involves establishing a universal damage calculation model (DCM) [48]. The scholar pointed out

that various non-invasive spectral technologies (such as FTIR, hyperspectral imaging, etc.) can bring new perspectives to the evaluation of lighting damage to materials [48, 49]. The lighting damage evaluation method proposed in this study can be used to establish a DCM, whose key lies in the spectral responsivity function of the two components of substrates and colorants. The limitations of color difference for evaluating lighting damage to substrates can be addressed by the proposed method of FTIR combined with PCA. However, the color difference as a commonly used method for colorants also has well-documented limitations, such as abnormal hue angle shifts [48], and transformation needs before interpreting and comparing the results in different studies using different color difference formulas [13, 50]. In fact, the reflection spectrum of a material reflects its physical property in terms of spectral selective absorption and reflection of incident light, which is the basis for color measurement and calculation such as color difference [51]. The reflection spectrum, as the “fingerprint” spectrum of the color of colorants, fundamentally determines the color change and can be used to accurately identify its color damage. It is feasible to evaluate the lighting damage to colorants using a spectral quantification method combining reflection spectra with PCA in the subsequent studies. It is worth noting that since the late 1990s, micro fading spectroscopy has been developed to evaluate the stability of colorants. On that basis, the reflection spectrum can be effectively measured to evaluate the lighting damage in a very short time, which is a significant advantage compared to traditional accelerated aging experiments that typically require longer time [52].

Conclusions

In this study, based on the accelerated aging experiment of nine narrowband LEDs in the visible range on silk samples with different acidification degrees, the infrared spectrum was measured non periodically, whose data processing was carried out with PCA. The ΔPC_1 calculated was used to evaluate the lighting damage. Based on the derivation of the damage rate, the damage inflection point and the corresponding exposure were determined. On that basis, the spectral responsivity of silk substrates with different acidification degrees was quantized to obtain its function, namely, the mathematical relationship between lighting damage and λ and pH values. In combination with the previous study on the spectral responsivity of colorants, an evaluation method for lighting damage was proposed, which is the key to the conservation of calligraphy and painting relics.

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Author contributions

ZW was the project leader and was a major contributor in writing the manuscript. ZF performed the acquisition, analysis, and interpretation of data. HT designed the work and substantively revised the manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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