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Matching Dyeing and Properties of Silk Fabrics with Natural Edible Pigments

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Abstract: The silk fabrics were matching dyed with three natural edible pigments (red rice red, ginger yellow and gardenia blue). By investigating the dyeing rates and lifting properties of these pigments, it was observed that their compatibilities were excellent in the dyeing process; dye dosage 2.5% (omf), mordant alum dosage 2.0% (omf), dyeing temperature 80 °C and dyeing time 40 min. The silk fabrics dyed with secondary colors exhibited vibrant and vivid color owing to the remarkable lightness and chroma of ginger yellow. However, gardenia blue exhibited multiple absorption peaks in the visible light range, resulting in significantly lower lightness and chroma for the silk fabrics dyed with tertiary colors, thus making it suitable only for matte-colored fabrics with low chroma levels. In addition, the silk fabrics dyed with these three pigments had a color fastness that exceeded grade 3 in resistance to perspiration, soap washing and light exposure, indicating acceptable wearing properties. The dyeing process described in this research exhibited a wide range of potential applications in matching dyeing of protein-based textiles with natural colorants.

Key words: matching dyeing; silk fabric; natural edible pigment; secondary color; tertiary color

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0 Introduction

Natural dyes, derived from plants, minerals and even insects, have been employed in textile production for centuries^[1-4], offering a sustainable and environmentally friendly alternative to synthetic counterparts. The use of natural dyes not only adds vibrant and distinct hues to fabrics but also aligns with the growing global emphasis on eco-conscious practices in various industries^[5-8]. Extracted from plants, dyes like indigo^[9-12], madder^[13-15] and turmeric^[16-18] not only give a diverse color palette to textiles but also contribute to a reduction in the environmental impact associated with

traditional dyeing processes. While natural dyes have gained popularity due to their eco-friendly and sustainable qualities, they also suffer certain drawbacks when applied in the textile industry, such as limited color range and color fastness, inconsistency in color and high cost, which limit their commercialization in textiles.

Natural edible pigments can be employed as colorants for various food products, offering exceptional safety, while also providing diverse nutritional and pharmacological benefits^[19-22]. In comparison to natural dyes utilized in textile dyeing processes, the demand for natural edible pigments is significantly higher, resulting in elevated levels of industrialization and relatively consistent product performance^[23]. The utilization of natural edible pigments as colorants for textiles is commonly found in literature reports^[5,13,24-27], albeit primarily limited to monochrome or secondary color combinations. This restriction arises from the varying affinities and diffusion rates exhibited by different natural pigments toward fibers. When pigments with significantly divergent dyeing rates are dyed on fabrics, they result in uncontrollable color hues and uneven dyeing. Consequently, reported instances are scarce regarding the matching dyeing of textiles using natural edible pigments, particularly those involving tertiary colors.

The present study focused on the selection of red rice red, ginger yellow and gardenia blue as the three primary colors for dyeing silk fabrics. Firstly, the dyeing rates and lifting properties of these pigments were studied to optimize the dyeing process and verify their compatibility. Subsequently, the effects of matching ratios on K/S value, lightness L^* , red-green degree a^* , yellow-blue degree b^* , chroma value C , and hue value H of dyed silk fabrics were investigated by matching of red and yellow, yellow and blue, as well as blue and red, respectively. In addition, the properties of secondary and tertiary colors and the corresponding application conditions for dyed silk fabrics were clarified by comparing the color characteristic values. Finally, measurements were performed on the color fastness of the dyed silk fabrics.

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1 Materials and Methods

1.1 Materials

The silk twill fabrics (140×140 , 50 g/m^2) were purchased from Zhejiang Xidebao Silk Technology Co., Ltd., China. Red rice red, ginger yellow and gardenia blue were obtained from Zhejiang Nuoyi Biotechnology Co., Ltd., China, and purified before use. The chemical structures of the main components of the three natural edible pigments are depicted in Fig. 1. The mordant alum ($\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, purity > 99%) and acetic acid were purchased from Shanghai Aladdin, China.

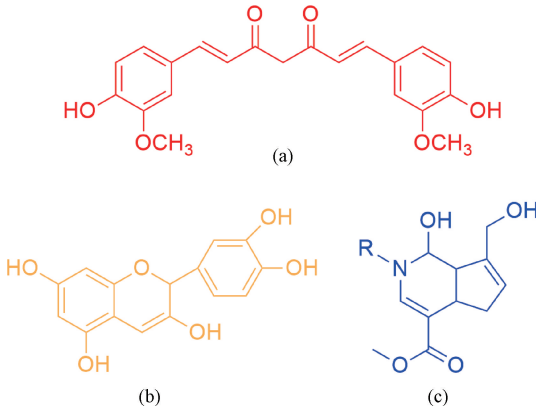


Fig. 1 Chemical structures of the main components of three natural edible pigments: (a) red rice red; (b) ginger yellow; (c) gardenia blue

1.2 Methods

1.2.1 Dyeing process

The dye liquor was prepared by blending dyes (2.0% (omf)) and mordants (2.0% (omf)) with a bath ratio of 1:30. The pH value of the dye liquor was adjusted to 4–5. The dyeing process was carried out in an infrared dyeing machine (HWX-24, Quanzhou Meibang Instrument Technology Co., Ltd., China). The dyeing temperature increased from room temperature to $80 \text{ }^\circ\text{C}$ at a heating rate of $1 \text{ }^\circ\text{C}/\text{min}$. After dyeing for 40 min, the fabrics were taken out and washed with the saponin solution (2 g/L , $60 \text{ }^\circ\text{C}$), then rinsed with water and dried.

1.2.2 Matching of secondary and tertiary colors

Matching of secondary colors. The combination of two dyes mixed in mass ratios of 10:0, 9:1, 7:3, 5:5, 3:7, 1:9 and 0:10 were selected, thus each secondary color consisted of seven samples, resulting in a cumulative total of 18 samples after eliminating three duplicate samples.

Matching of tertiary colors. An $L_{25}(5^6)$ orthogonal array with three factors (red, yellow and blue) and five levels (1, 3, 5, 7 and 9) was constructed. Then the ratios were normalized to calculate the appropriate amount of dye for each sample. The mass ratios of red rice red : ginger yellow : gardenia blue are: 1:1:1, 1:3:3, 1:5:5, 1:7:7, 1:9:9, 3:1:3, 3:3:5,

3:5:7, 3:7:9, 3:9:1, 5:1:5, 5:3:7, 5:5:9, 5:7:1, 5:9:3, 7:1:7, 7:3:9, 7:5:1, 7:7:3, 7:9:5, 9:1:9, 9:3:1, 9:5:3, 9:7:5 and 9:9:7.

1.2.3 Dyeing uptake and lifting of dyes

The absorbance of the dye liquor was measured using an ultraviolet (UV)-Vis spectrophotometer (UV-5500PC, Shanghai Metash Instrument Co., Ltd., China) both before and after dyeing. The dye uptake D_u and dye amount D_a are calculated by

$$D_u = (1 - A/A_0) \times 100\%, \quad (1)$$

$$D_a = (1 - A/A_0)C, \quad (2)$$

where A_0 and A represent the absorbance at the maximum wavelength of the dye liquor before and after dyeing, respectively; C is the amount of dye depending on the mass of the silk fabrics before dyeing.

1.2.4 Color characteristic value

The color characteristic values of the silk fabrics were measured with a spectrophotometer (Datacolor 800, Datacolor Co., USA) equipped with a D65 light source and a spectral scanning range of 350–800 nm.

1.2.5 Color fastness

The color fastnesses were evaluated according to GB/T 3921—2008 *Textiles—tests for color fastness—color fastness to washing with soap or soap and soda*, GB/T 3922—2013 *Textiles—tests for color fastness—color fastness to perspiration* and GB/T 8427—2019 *Textiles—tests for color fastness—color fastness to artificial light: Xenon arc fading lamp test*.

The silk fabrics treated according to the above testing standards were rated referring to GB/T 250—2005 *Textiles—tests for color fastness—gray scale for assessing change in color*.

2 Results and Discussion

2.1 Compatibility of dyes

The compatibility of dyes is crucial for color matching. We assessed the color-matching performance of three natural edible pigments, namely red rice red, ginger yellow and gardenia blue, by measuring their dyeing uptake and lifting of dyes at various temperatures.

As depicted in Fig. 2, all three pigments are rapidly dyed on silk fabrics during the initial stage. Because the three pigments contain a large number of hydroxyl or carbonyl groups, in the presence of metal ions of the mordant, they can form hydrogen bonds and coordination bonds with carboxyl groups, amino groups and hydroxyl groups on the side chains of silk fibers. Meanwhile, the hydroxyl groups bear negative charges and can establish ionic bonds with the positive charges on silk fibers under acidic conditions. After dyeing for 40 min at $60 \text{ }^\circ\text{C}$, all three pigments reach a state of dyeing equilibrium with a dye uptake of approximately 80%. The order of the dye affinity is as follows: red rice red > ginger yellow >

gardenia blue. This indicates that these three pigments exhibit a favorable affinity towards silk fabrics in the presence of the mordant. Moreover, when the dyeing temperature increases to 80 °C, the dye uptake of all three pigments is significantly enhanced, achieving approximately 95%. The temperature rise enhances the kinetic energy of dye molecules and increases the pore size of the fibers, thereby facilitating dye penetration into the fiber interior and improving the dye uptake. For these three pigments, a high dye uptake has already been achieved at 80 °C. However, exceeding this temperature may potentially impact the structure of natural dyes. Therefore, 80 °C is an optimal temperature for dyeing silk fabrics with these three pigments.

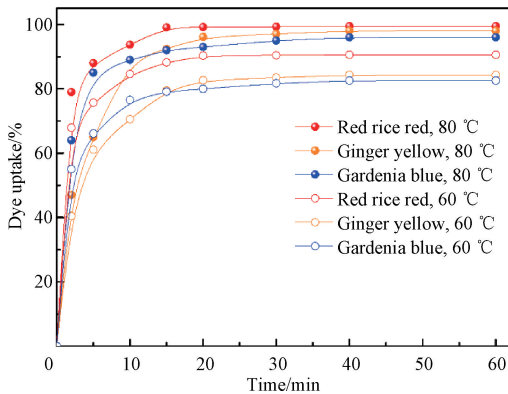


Fig. 2 Dye uptake of red rice red, ginger yellow and gardenia blue as a function of dyeing time at the temperature of 60 °C and 80 °C

By varying the dosage of these three pigments, a comparison was made on the differences in the dye amount on fabrics between 60 °C and 80 °C. As illustrated in Fig. 3, there is an almost linear increase in the dye amount with the increasing dye dosage below 1.0% (omf) at 60 °C. Further increasing the dye dosage, the dye amount for all fabrics reaches equilibrium at approximately 2.5% (omf) and shows a minimal change when the dye dosage increases to 3.0% (omf). The results demonstrate that the silk fabrics reach saturation in terms of adsorption for all three pigments at a dosage of 2.5% (omf). Among them, red rice red exhibits the highest dye amount due to its longer conjugated system in the molecular structure, which enhances its affinity to silk fibers, compared to the other two pigments. By increasing the dyeing temperature to 80 °C, a corresponding enhancement in the equilibrium of the dye amount for all three pigments is achieved.

Based on the discussion above, it is found that the optimal dyeing process of red rice red, ginger yellow and gardenia blue on silk fabrics are as follows: dye dosage 2.5% (omf), mordant alum dosage 2.0% (omf), dyeing temperature 80 °C and dyeing time 40 min. The matching dyeing on silk is suitable for these three natural pigments under the given dyeing condition.

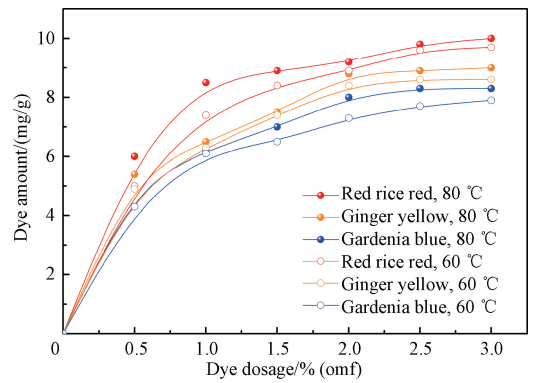


Fig. 3 Dye amount of red rice red, ginger yellow and gardenia blue as a function of dye dosage at the temperature of 60 °C and 80 °C

2.2 Matching of secondary colors

The scatter plots are generated by utilizing the mass ratio of secondary color, resulting in the formation of a triangle in three dimensions. The data points are uniformly distributed along the edges of this triangle (Fig. 4(a)). From Figs. 4(b)–4(f), it is evident that the varying mass ratios of these three pigments exert a substantial influence on the K/S , hue, chroma and lightness values of silk fabrics. In Fig. 4(d), the maximum peak absorption wavelength at 525 nm characterizes monochromatic red. With an increasing mass ratio of ginger yellow, there is a gradual decline in the K/S value. At a mass ratio of 5 : 5, there is an inversion in the maximum peak absorption wavelength, resulting in 430 nm. Simultaneously, a^* decreases while b^* increases and L^* improves (Fig. 4(b), R, G and B in Fig. 4(b) represent red, green and blue, respectively), indicating a transition from red to yellow and an improvement in the lightness of the silk fabrics. Subsequently, there is a gradual increase in the K/S value and a change in the hue value of the fabrics from 14° to 60° while chroma remains relatively unchanged (Fig. 4(c)). Further increasing the mass ratio of ginger yellow leads to an additional enhancement in the lightness and the chroma of silk fabrics. Moreover, when the mass ratio exceeds 5 : 5, an upward trend can be observed in K/S values. The results suggest that both red rice red and ginger yellow have a comparable effect on the K/S value and the chroma of silk fabrics dyed with secondary colors, whereas ginger yellow significantly enhances the lightness of silk fabrics.

The absorbance curves of the silk fabrics dyed with ginger yellow and gardenia blue are depicted in Fig. 4(e). Ginger yellow exhibits its maximum absorption wavelength at 425 nm, resulting in a low K/S value of 2.5 but high chroma and lightness. When the mass ratio reaches 7 : 3, the dominant color shifts to gardenia blue. As the mass ratio of gardenia blue increases further, the lightness of the silk fabrics gradually decreases while the K/S value rapidly increases due to the narrow and sharp absorption peak at 625 nm. It

is worth noting that the chroma values of the fabrics also demonstrate a decreasing trend because gardenia blue has an additional absorption peak at 425 nm (Fig. 4(e), orange hollow square). The presence of multiple absorptions result in the formation of mixed complementary colors, leading to a decrease in the chroma for gardenia blue^[28]. Conversely, ginger yellow exhibits a broad absorption peak solely at 425 nm within the visible light range^[29], contributing to its complementary color appearing as highly bright and vibrant hues.

Figure 4(f) illustrates the absorbance curves of the silk fabrics dyed with gardenia blue and red rice red. Due to the significant absorption peak at 625 nm of gardenia blue, red rice red dominates the secondary color until reaching a mass ratio of 3 : 7. Subsequently, while the *K/S* value of the silk fabrics gradually increases, it remains noticeably lower than that observed for gardenia

blue. Additionally, the lightness and the chroma of the silk fabrics decrease with an increasing mass ratio of gardenia blue. The curves possess distinct peaks at 425, 525 and 625 nm, resulting in a more blended complementary hue and consequently reducing chroma to its minimum level.

Comparing these three pigments, it can be observed that ginger yellow exhibits higher levels of lightness and chroma while having a lower *K/S* value. In contrast, gardenia blue displays lower levels of lightness and chroma but has a higher *K/S* value. Red rice red falls somewhere in between. Considering the overall secondary color situation, the lightness values range from 51.9% to 91.2%, hue values span from 14.1° to 347.7°, and chroma values ranges from 13.3% to 65.5%. Through the analysis of the matching of secondary colors, it can be inferred that silk fabrics dyed with these three pigments exhibit vivid and vibrant hues.

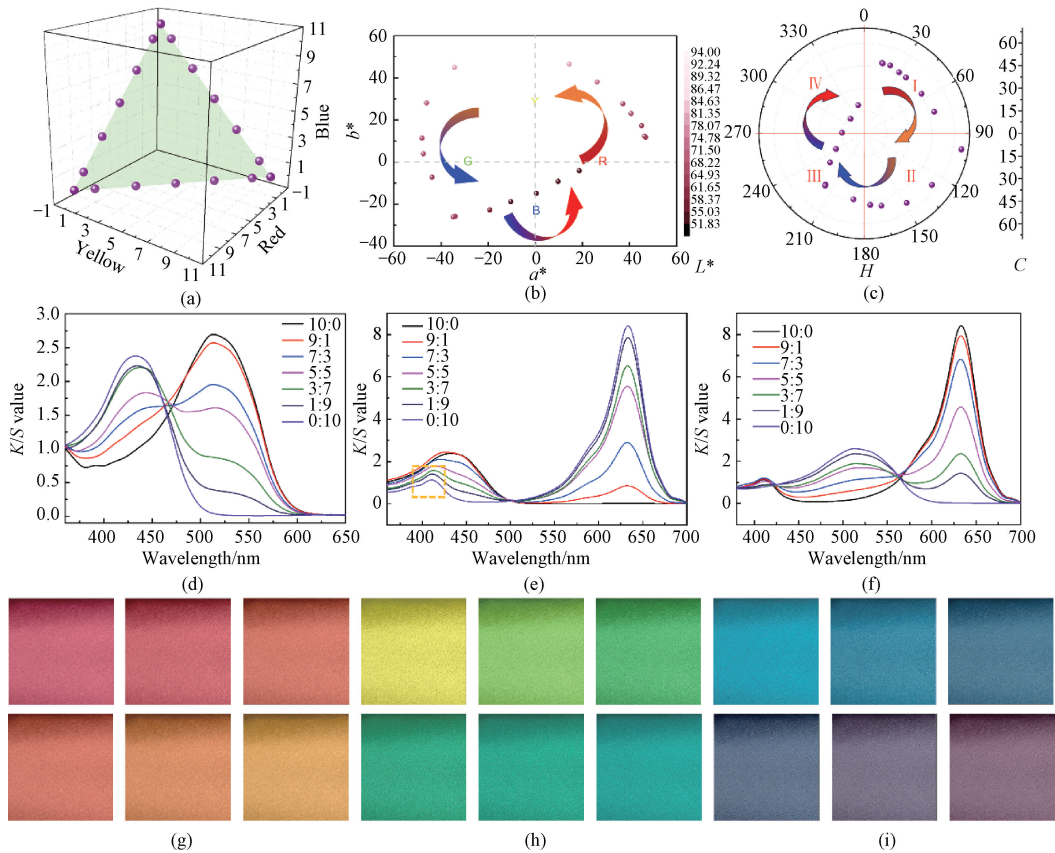


Fig. 4 Characteristic values and images of secondary colors: (a) scatter plots for the mass ratios of secondary colors; (b) L^* , a^* and b^* of silk fabrics dyed with secondary colors; (c) C and H of silk fabrics dyed with secondary colors; (d) K/S value as a function of wavelength for silk fabrics dyed with red rice red and ginger yellow; (e) K/S value as a function of wavelength for silk fabrics dyed with ginger yellow and gardenia blue; (f) K/S value as a function of wavelength for silk fabrics dyed with gardenia blue and red rice red; (g)–(i) images of silk fabrics corresponding to (d)–(f) sequentially

2.3 Matching of tertiary colors

The scatter plots are constructed with the matching ratio of tertiary colors (Fig. 5(a)). Following the principle of orthogonal design, the sampling points are

categorized into five groups. By connecting these sampling points to five polygons, it can be observed that each color occurs in equal proportions, indicating a uniform dispersion of sampling points in this experiment.

This effectively covers the matching of three colors.

Compared to secondary colors, tertiary colors demonstrate a greater range of diversity within the $L^*a^*b^*$ color space. However, their lightness values fall within a range of 48.9% to 67.1% (Fig. 5(b)), which is noticeably lower than that of secondary colors (51.9% – 91.2%). This discrepancy arises from the principles of subtractive color mixing in dye matching, where an increase in dye variety leads to enhanced absorption of natural light across various wavelengths and consequently diminishes reflected light, resulting in decreased lightness. In addition, from the perspective of chroma and hue values, the hue values of tertiary colors are

mainly concentrated between 0° and 270° (Fig. 5(c)), which correspond to the region between red and purple. These hue values exhibit a uniform distribution across regions I, II and III, mirroring that of secondary colors. However, the chroma values of tertiary colors exhibit a color variation range of 5.0% to 34.7%, with the majority falling below 20%. This low chroma of tertiary colors can be attributed to more multiple absorptions compared to secondary colors. No matter what, the incorporation of tertiary colors can effectively address the deficiencies in hue found in secondary colors and fulfill the requirements for achieving low chroma matte color (Fig. 5(d)) on silk fabrics.

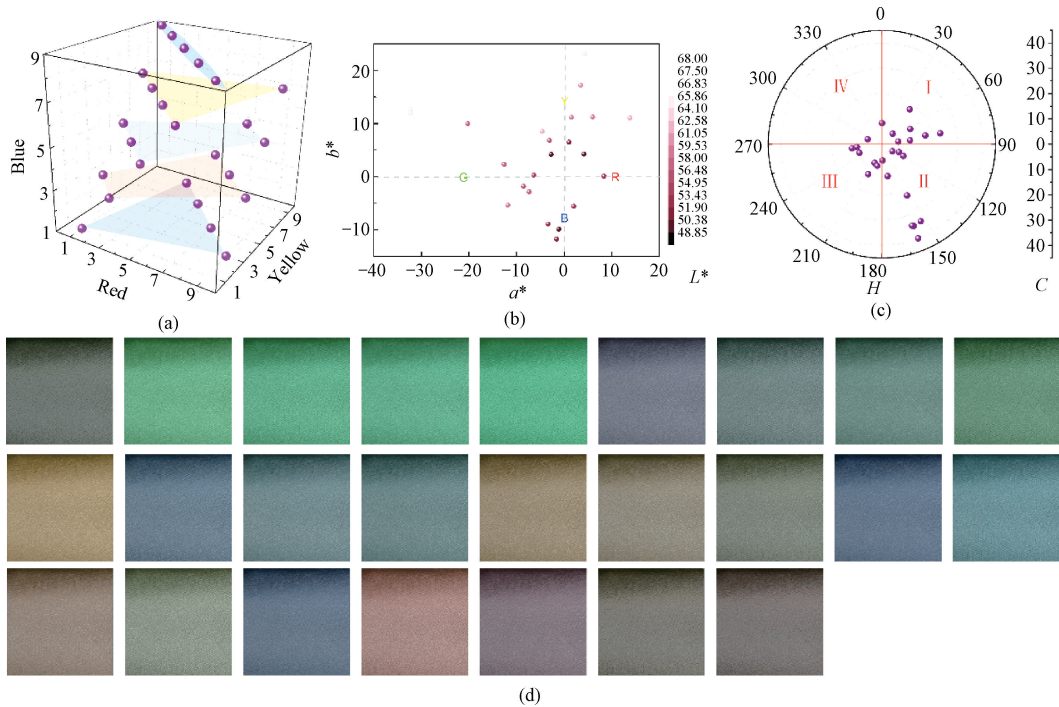


Fig. 5 Characteristic values and images of tertiary colors: (a) scatter plots for the mass ratios of tertiary colors; (b) L^* , a^* and b^* of silk fabrics dyed with tertiary colors; (c) C and H of silk fabrics dyed with tertiary colors; (d) images of silk fabrics dyed with tertiary colors

2.4 Color fastness

The selected silk fabrics dyed with tertiary colors were subjected to test for color fastness to perspiration, soap washing and light exposure. The corresponding results are presented in Table 1. R : Y : B in Table 1 represents the mass ratios of red rice red : ginger yellow : gardenia blue.

The color fastness of silk fabrics all exceeds grade 3, demonstrating an acceptable color fastness. For detailed analysis, it can be observed that when the mass ratio of red rice red is high, the color fastness is decreased, especially for the soap-washing fabric. The possible reason for this is that red rice red has a relatively high molecular weight, making it

difficult to penetrate the interior of the fiber during the dyeing process. As a result, the dye on the surface is easier to detach from the fabrics during soap washing. Nevertheless, the silk fabrics dyed with natural pigments satisfy the requirements for daily wear. This can be attributed to the relatively good stability of the selected three pigments and their strong affinity with silk fibers. Furthermore, the addition of a mordant facilitates the formation of ionic bonds between metal cations, pigments and fibers, thereby improving light fastness as well. Therefore, silk fabrics dyed with a matching color of red rice red, ginger yellow and gardenia blue can effectively meet textile color fastness requirements.

Table 1 Color fastness of silk fabrics dyed with tertiary colors

R : Y : B	Perspiration			Soap washing			Light exposure
	Discoloration	Staining (silk)	Staining (cotton)	Discoloration	Staining (silk)	Staining (cotton)	
1 : 1 : 1	3-4	4-5	4	4-5	4	4	3
1 : 3 : 3	3-4	4	4	4	4	4	3-4
3 : 1 : 3	4	4-5	4	3-4	4	3-4	3
3 : 5 : 7	4	4	4-5	4	3-4	4	3
5 : 1 : 5	3-4	4	4	3-4	4	4	3
5 : 5 : 9	4	3-4	4	3-4	4	3-4	3-4
7 : 9 : 5	3	4	4	3	3-4	4	3
7 : 3 : 9	4	4-5	5	4	4	3-4	3-4
9 : 1 : 9	3-4	4	4	4	3	3	3
9 : 5 : 3	4	4	3-4	3	3	3	3

3 Conclusions

Three natural edible pigments, namely red rice red, ginger yellow and gardenia blue, were applied to the matching dyeing of silk fabrics. The optimal dyeing process of the pigments on silk fabrics is as follows: dye dosage 2.5% (omf), mordant alum dosage 2.0% (omf), dyeing temperature 80 °C and dyeing time 40 min. Under this condition, the dyeing uptake and lifting of dyes of these three pigments exhibit a high degree of similarity. Comparing these three pigments, it can be observed that ginger yellow exhibits higher levels of lightness and chroma while having a lower K/S value. In contrast, gardenia blue displays lower levels of lightness and chroma but has a higher K/S value. Red rice red falls somewhere in between. Considering the overall secondary color situation, the lightness values range from 51.9% to 91.2%, hue values span from 14.1° to 347.7°, and chroma values range from 13.3% to 65.5%. The silk fabrics dyed with secondary colors exhibit vivid and vibrant hues. Tertiary colors demonstrate a greater range of diversity within the $L^*a^*b^*$ color space. However, their lightness values noticeably decrease. Nevertheless, tertiary colors can effectively address the deficiencies in hue found in secondary colors and fulfill the requirements for achieving low chroma matte color on the silk fabrics. The silk fabrics dyed with these three pigments can effectively meet textile color fastness requirements. The application and the promotion of this dyeing technique are highly commendable.

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丝织物的天然食用色素拼色染色及性能研究

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摘要: 该研究选用三种天然食用色素(红米红、姜黄和栀子蓝)拼色染色丝织物。通过三种色素的上染速率与提升率研究发现,在色素用量为 2.5% (omf),媒染剂明矾用量为 2.0% (omf),染色温度为 80 ℃,染色时间为 40 min 的条件下,三种色素的配伍性良好。由于姜黄色素的亮度与饱和度较高,因此二次色染色丝织物可呈现鲜艳且明亮的颜色。由于栀子蓝在可见光范围内具有多处吸收峰,导致三次色染色丝织物的亮度和饱和度较二次色明显降低,只能适用于低饱和度的哑光色织物。三次色染色丝织物的耐汗渍、皂洗和耐光牢度均大于 3 级,表明通过上述三种色素染色的丝织物具有良好的色牢度。该染色工艺在天然色素拼色染色蛋白质类纺织品方面具有广阔的应用前景。

关键词: 拼色染色; 丝织物; 天然食用色素; 二次色; 三次色