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Effects of yellow natural dyes on handmade Daqian paper

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Abstract

Natural yellow plant dyes and traditional medicines were used widely on historical papers in ancient China for religious reasons and conservation considerations. This study aims to evaluate some traditional yellow botanical sources of dyes that contain different chemical colorant compositions in order to understand their effects on the properties of traditional handmade paper. The physical and chemical changes in paper specimens treated with plant dyes were studied by examining properties such as the color, pH, thermogravimetric (TG) characteristics, tensile strength, folding endurance and microstructure by scanning electron microscopy (SEM). The results indicated that different colorants had different toning effects and that the main components, including carboxyl and ketone groups, could affect the paper stability at high temperatures. The results also revealed that the mechanical properties of paper specimens were improved after treatment with plant dyes. The significant improvements in the tensile strength and folding endurance and the slightly higher decomposition temperature of *Amur cork tree*-dyed paper could be ascribed to the strong interaction between the colorants' main components and the fibers. The scientific evaluation of the property changes is therefore valuable information for weighing the advantages and disadvantages of the various yellow toning materials for paper conservation treatment.

Keywords: Yellow natural dyes, Property changes, Historical paper, Conservation

Introduction

Throughout history, botanically-sourced dyes extracted from locally available plants have been used to color papers, textiles and other objects. According to the remaining colored ancient Chinese books, yellow dyes have been widely used for thousands of years. The most famous of these books are the large collections of ancient Buddhist manuscripts at *Dunhuang*, some of which were dyed yellow [1, 2]. There are many reasons for the use of plants rich in yellow colorants in China: religious purposes (yellow represents solemnity in Buddhism); in hierarchical symbols because yellow was regarded as an imperial color—the emperors and imperial family of China were the only ones allowed to wear yellow robes; the high tinctorial strength of these plant dyes; the vividness of the colors obtained from them (even though some are not very colorfast); their ease of use; and last, conservation considerations, since yellow alkaloid dyes have insecticidal properties; that is, berberine, curcumin, crocetin and rutin dyes can be used as antioxidants and antibacterial materials. Even the Chinese government decree of AD 675 stated that yellow paper should be used by various government offices when issuing decrees and orders since white paper was often damaged by insects [3].

The yellow or golden color of autumn leaves, roots and tree bark are important sources of yellow dyes and paints. Since the earliest times, dyers worldwide have discovered a wealth of yellow dyes in many botanical sources. The pagoda bud (called *huaimi* in Chinese, the pagoda flower and bud of *Sophora japonica* L.), gardenia (called *zhizi* in Chinese, the fruit of *Gardenia jasminoides Ellis*), turmeric (called *qianhuang* in Chinese,

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the rhizome of *Curcuma longa* L.), and the Amur cork tree (called *huangbo* in Chinese, the inner part of the bark of *Phellodendron amurense Rupr.*), were known and used for thousands of years in China. The dyes can be fixed onto paper fibers by a brushing or pulling process without the need for a mordant, a quality that makes them quite easy to use and that, along with their fair anti-insect and/or colorfastness properties, has ensured their long-lasting popularity with dyers. A description of how to use the pagoda bud for dyeing was included in the eighth century *Bencao shiyi* [4]. The cultivation and use of gardenia for dyeing have been recorded since the Northern and Southern Dynasties (420–589 AD) [5]. Turmeric was recorded as a yellow dye in historical documents such as *Bencao gangmu* (本草纲目, *Compendium of Materia Meica*, 1578)[6]. A detailed description of the preparation of Amur cork tree dye for dyeing paper was found in *Qiming yaoshu (*齐民要术*, Main techniques for the welfare of the people, ca. 533–544 AD)*, written during the Northern Dynasty (386–534 AD)[7]. Multidisciplinary research involving botanists, historians and chemists has already yielded interesting results, showing that these yellow dye plants contain different color sources [8]. Gardenia contains crocin and crocetin colorants and produces colors ranging from yellow to orange-red. Turmeric contains curcumin and provides a bright yellow color. Amur cork tree contains the colorant berberine, a kind of bright yellow material. The main colorant in the pagoda buds from opened/unopened pagoda trees has been identified as rutin. In China, all of these dyes could be used directly without mordant dyeing papers. Usually, dried botanical sources are thoroughly soaked, pounded, boiled and pressed in a cloth sack. The prepared liquid is saved and heated to a certain temperature to dye paper by a brushing or pulling process.

As an increasing number of historical papers have been discovered that have been dyed with different botanical yellow dyes, learning how these plant dyes affect the properties of traditional handmade papers is of interest. Paper conservators and museum professionals will be able to make better decisions when selecting colorants for conservation treatment. There have been some reports about natural yellow dyes used in ancient objects [9–13]; unfortunately, very few reports on ancient paper properties have been published prior to now. Considering the many works on natural yellow dyes, a strong project might result in several papers. In the present study, we limited our research to comparing the properties of handmade paper dyed with four different natural yellow dyes. To avoid contamination by other materials, no mordant was used during the dyeing process.

Experimental

Materials

Handmade Daqian paper (25.46 g/m^2 in grammage and 94 µm in thickness), in which the paper pulp was not bleached, was obtained from the *Daqian* Paper Shop in Sichuan Province.

The gardenia, pagoda bud, turmeric, and Amur cork tree materials were purchased from a local Chinese medicine store in Chengdu. All materials were purchased commercially and used as received. Photographic images of these botanical sources are presented in Fig. 1.

Preparation of colorant dyes

The dyeing process for botanical sources usually includes sourcing botanical plants, extracting dyeing components and dyeing while controlling parameters such as temperature and time duration in dyeing experiments. In our experiments, the dyestuff extraction procedure was based on traditional Chinese recipes adopted for laboratory procedures, and a liquor ratio of 1 g of plant material to 20 mL deionized water was maintained [14–16]. Commercially available plant materials (50 g) were chopped into small pieces and soaked at room temperature in 500 mL of deionized water for a period of 12 h, followed by gradually heating the mixtures to boiling and simmering them at 80 ℃ with regular stirring for 30 min. Then, the liquor was obtained by sedimentation and mixed with pure juice pressed from the boiled dregs in a piece of cloth sack. This procedure was repeated twice to allow as many of the colorants as possible to come out of the plants. Both times, as much liquid as possible was mixed and saved for later use. Photographic images of the prepared colorant dyes are presented in Fig. 2.

Preparation of the dyed paper samples

The dyed paper samples were prepared by pulling papers in different temperature colorant dyes according to traditional Chinese recipes and the stability and solubility of different colored compound: the colorant dyes were kept at 40–50 ℃ when dyeing with the gardenia and pagoda bud dyes, at 50–60 ℃ with the turmeric dye, and at 80–90 ℃ with the Amur cork tree dye. The treated papers were hung on glass rods at 20–25 ℃ for 48 h. These dyed paper samples were labeled YP-A, YP-P, YP-G, or YP-T, where A, P, G, and T indicated the plant dyes of the Amur cork tree (A), pagoda bud (P), gardenia (G) and turmeric (T). The untreated paper samples were labeled YP-0. Photographic images of the colorant dyed papers are presented in Fig. 3.

Fig. 1 Photographic images of botanical sources of yellow natural dyes

Characterization

pH tests of colorant dyes

Portable pH measurements (Horiba, LAQUA twin-pH-22) were used to test the pH of the prepared colorant dyes. The light shield cover was opened, and 3–4 drops of liquid colorant dyes were placed on the flat sensor to cover the entire flat sensor. The dye was allowed to rest on the sensor until the measured value was displayed.

pH tests of papers

The pH tests of the papers were carried out under laboratory conditions using a cold extraction measurement, which is more accurate than a surface method but also more destructive. For this method, 1 g of paper samples was cut into small fractions and left in 100 ml of cold, distilled water for 1h. Then, the pH of the cold extract was analyzed without filtration under stirring using a Mettler Toledo Inlab Power pH meter (Mettler Toledo, Switzerland) according to the standard ISO 6588–1:2012 [17]. The accuracy of the pH measurements was an average pH < ±0.02 units (n = 5).

Weight change measurements of paper

The percentage of weight change of the paper was calculated from the following equations. All the values presented are the average of three specimens.

$$\text{Weight change } (\%) = 100 \times \frac{\mathrm{W}_d - W_0}{W_0} \tag{1}$$

where W_d is the weight of dyed paper and W_0 is the weight of undyed paper.

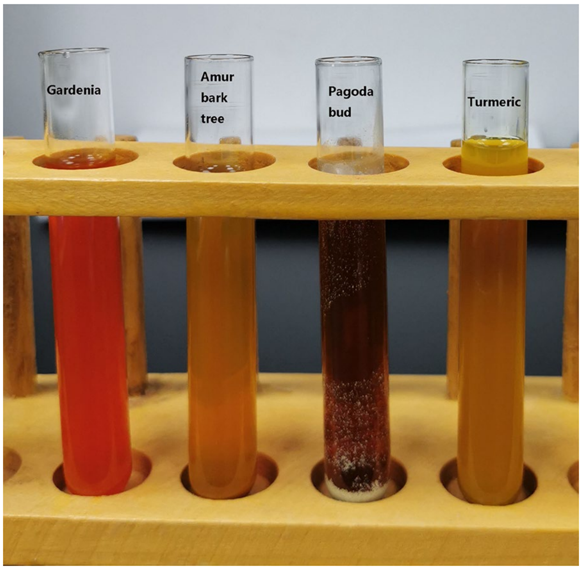

Fig. 2 Photographic images of prepared colorant dyes

Color change measurements of paper

The change in the color of the paper was determined using a solid reflection spectrophotometer (CM-700D from Minolta Co., Japan) according to the standard ISO 11475:2004 [18]. The conditions used in the experiment were the standard illuminant D65 and the 10° observer. The CIEL*a*b* color space was used, and the color variations were evaluated using the parameter total color difference (ΔE*). The samples were always measured at five identical places. The average color difference was expressed as $\Delta E = \sqrt{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2}$.

ATR-FTIR analyses

Infrared spectra were obtained with a Thermo Nicolet iZ10 FTIR (Thermo Scientific Instrument) spectrophotometer combined with a Smart Orbit single reflection diamond attenuated total reflectance (ATR) accessory from 4000 to 650 cm^{-1} for 128 scans with 4 cm^{-1} spectral resolution. The FTIR microscope was equipped with an internal pressure sensor, a very precise and accurate motorized X–Y stage, and an MCT detector and was cooled with liquid nitrogen.

Thermal analysis

Thermogravimetric (TG) analysis was carried out with a TGA 550 (Thermal Analysis Instrument). The samples were heated from room temperature to 900 °C at a rate of 10 °C/min in the TGA instrument under ultrahigh-purified nitrogen at a flow rate of 25 mL/min.

Mechanical property measurements

All the paper samples were conditioned according to ISO 187–1990 [19] before mechanical measurements at a temperature of 23 ± 1℃ and an RH of 50% ± 2% for 24 h.

The mechanical properties were determined according to the TAPPI and ISO standards. Tensile strength test specimens were prepared by cutting samples 15 mm wide with sides within 0.1 mm and 250 mm long in the horizontal and vertical directions. Tensile strength tests on dyed and undyed paper sheets were performed on a computer-controlled TMI 84–56 tensile tester (horizontal) (Testing Machines, Inc., Holland) at room temperature using an extensometer gauge of 180 mm × 15 mm, load cells of 100 N and a test speed of 25 mm/min. All tests were carried out according to the TAPPI T-494 and ISO

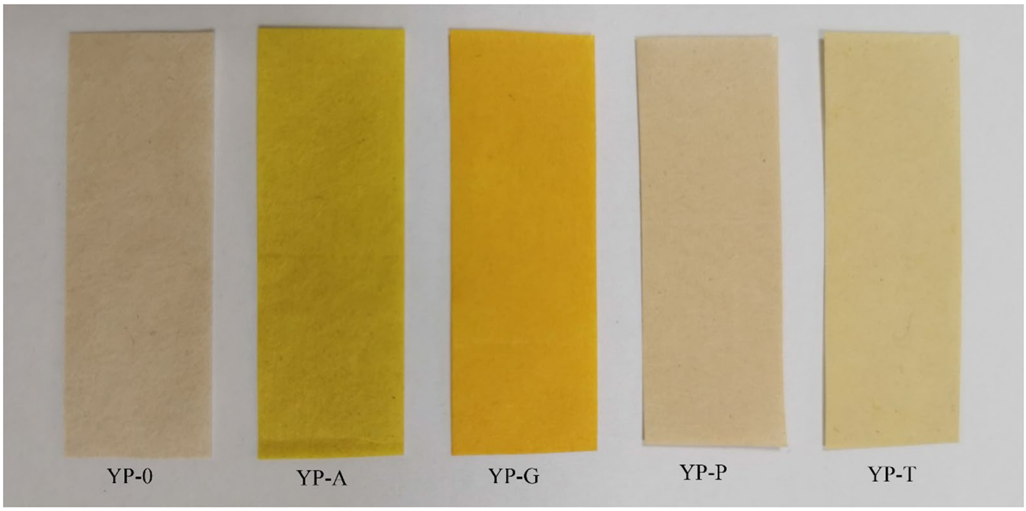

Fig. 3 Photographic images of dyed papers

Table 1 The colour changes of paper samples after dyeing with different botanical sources

Sample	**YP-0**	**YP-A**	**YP-P**	**YP-G**	**YP-T**
Color	**Yellowish**	**Bright yellow**	**Light yellow**	**Yellow to orange-red**	**Lemon yellow**
L	76.36	71.35	73.72	69.74	75.28
a	3.66	1.22	3.18	15.6	3.18
b	17.35	57.44	25.9	57.5	27.02
ΔE	–	40.48	8.97	42.28	9.74

1924 standards [20, 21]. The reported values were calculated as the averages of at least ten specimens.

The folding endurance experiments were performed on a TMI 31–23 folding endurance tester (Testing Machines, Inc., USA) according to TAPPI/ANSI T511 [22] and ISO5626:1993 [23] with standard 14 cm long by 15 mm wide paper samples. The applied force was 0.5 Kg and double folds were 175 per minute. The reproted values were calculated as the averages of at least ten specimens.

Microscope examination

Scanning electron microscopy (SEM) images were recorded with a Philips FEI INSPECT F instrument operated at 5 or 10 kV working voltage after specimen coating with a very thin gold layer deposited by sputtering under vacuum. The surface of the paper specimens was examined.

Results and discussion

Figures 1 and 2 show images of different dye plants and obtained liquid colorant dyes, respectively, under experimental conditions. In general, plant dyes caused highly visually perceptible color changes in the papers relative to their untreated counterparts [16]. The T dyes, G dyes, P dyes, and A dyes showed orange-red colors, yellow–brown colors, tea colors, and yellow–brown colors, respectively.

Colorimetric information was obtained by employing the International Commission on Illumination CIELAB color space, which mathematically simulates the perception of color by providing a standard process for measuring and quantifying color changes according to the standard test method (ISO11475:2004). The variables L*, A*, and B* of the International Commission on Illumination (CIE) color space are used to designate lightness-darkness, redness-greenness and yellowness-blueness, respectively. The variation in the colorimetric coordinate L*, a* and b* values is shown in Table 1. All dyed paper samples showed a reduction in lightness after dyeing with plant dyes, but the effects on redness and yellowness were different. The papers treated with A colorants experienced a slight reduction in redness and a strong increase in yellowness, which suggested that A had the greatest yellow effect on the paper samples. The color changes recorded for the G paper samples revealed an increase in both redness and yellowness but less yellowness than samples treated with A dyes, which suggested that the G dyed paper samples showed a significant red-yellow color. The paper samples treated with P and T dyes exhibited only a reduction in redness and a slight increase in yellowness, representing the smallest color change. The different color results demonstrated that the different dyes had different toning effects.

Table 2 PH results and weight changes of dyed papers

Sample	**YP-0**	**YP-A**	**YP-P**	**YP-G**	**YP-T**
pH	7.8	6.71	6.65	6.02	6.98
Weight change (%)	-	11.6	21.8	13.5	11.9

Table 3 PH results of colorant dyes

Botanical sources	**Amur cork tree**	**Pagoda bud**	**Gardenia**	**Turmeric**
pH	6.12	6.42	4.51	6.54

The pH results of the colorant dyes and paper samples are shown in Tables 2 and 3. According to Table 3, the undyed paper samples demonstrated weak alkalinity (pH = 7.8), which might be ascribed to the presence of alkaline materials during the papermaking process. After treatment with different plant dyes, the paper samples had a slightly lower pH than the undyed paper. By comparing Tables 2 and 3, it was found that the decreased pH of different dyed papers was induced by adding plant dyes that reacted with weak alkaline paper. Additionally, the different pH results and color changes demonstrated that the main components of these plant dyes were different. These results corresponded with reports that most plant dyes could decrease the pH of paper [24]. In the experiments, the G dyes exhibited the lowest pH. The main dye constituents of G include crocin and crocetin (shown in Fig. 4), both of which are yellow dyes with high tinctorial

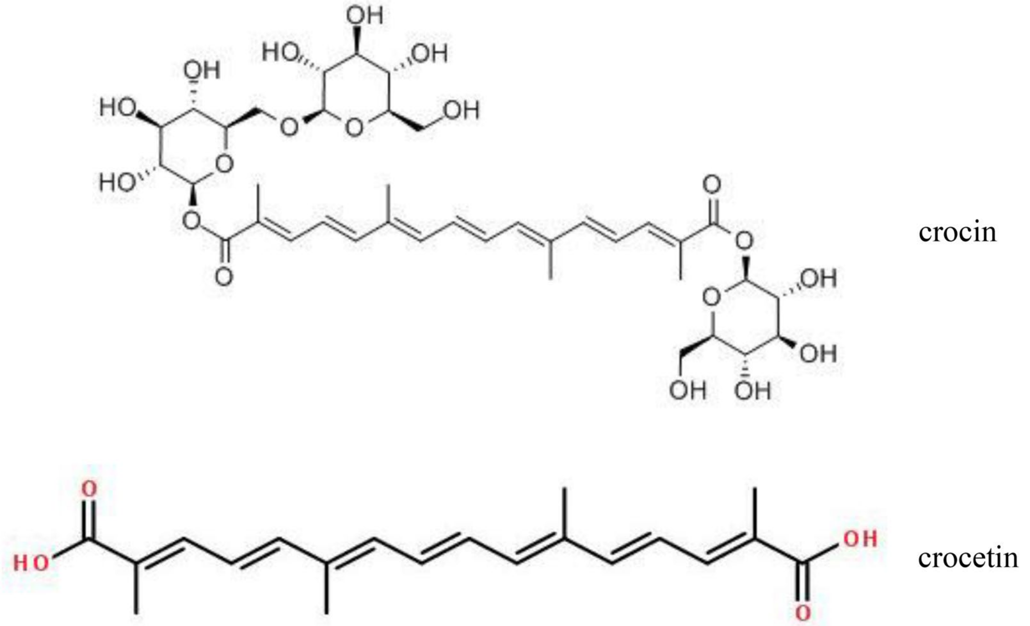

Fig. 4 The characteristic components of gardenia

strength and produce a golden-yellow hue on almost all materials. Crocin, with the molecular formula $C_{44}H_{24}O_4$ and a molecular weight of 997.21, contains natural glucoses on both sides of the molecule. Crocetin, with a molecular formula of $C_{20}H_{24}O_4$ and molecular weight of 328.4, contains carboxyl groups attached to both sides; these groups cause the G solution to have the lowest pH. It was reported that the carotenoid pigments extracted from G could be larger than those extracted from saffron [25]. However, G is quite unstable under light and alkaline conditions. For A, its main colored compound is berberine (shown in Fig. 5), with a strong yellow color and the molecular formula $C_{20}H_{18}NO_4$, which can be positively charged with protons to form a cationic alkaloid. The weak acidity might be due to the presence of obacunonic acid, ester, ketone and polysaccharide in solution. The main colored compound in T is curcumin (shown in Fig. 6). The phenol group in curcumin makes the T solution weakly acidic. Curcumin, which exhibits a yellow color and has a diferuloyl methane structure and a molecular weight of 368.4, was isolated in the nineteenth century [26]. Its colorant consists of a mixture of compounds known as curcuminoids. The main colorant in the P was rutin (shown in Fig. 7). Rutin, with the molecular formula $C_{27}H_{30}O_{16}$ and a molecular weight of 610.5, is a yellow crystalline flavonol glycoside that was isolated and identified by Stein in 1853 [27]. The four phenolic hydroxyl groups in rutin might be the reason why the solution is weakly acidic. Due to the different main components of the dyes, the dyed paper samples yielded different pH results, although all of the dyed papers had a slightly lower pH than the undyed papers. The presence of carotenoid pigments in G affected the pH of the dyed paper. To determine whether the pH change accelerates

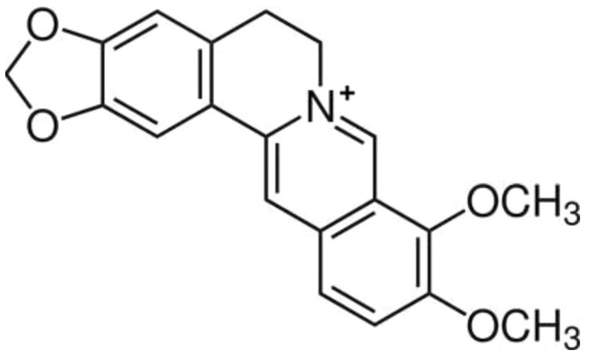

Fig. 5 Berberine, the characteristic components of Amur cork tree

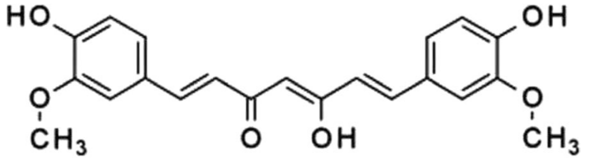

Fig. 6 Curcumin, the characteristic component of turmeric

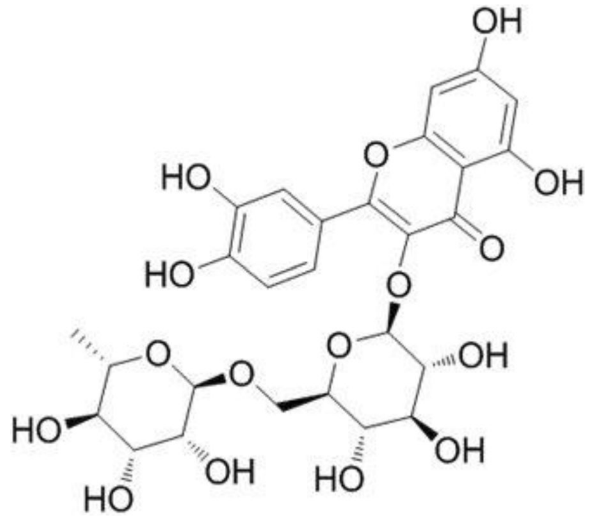

Fig. 7 Rutin, the characteristic components of pagoda bud

the instability of the paper during aging, we are conducting aging tests under thermal and UV-light artificial accelerating conditions. We will share our results in other reports in the near future.

Table 2 shows that the weight change of the paper samples was different after dyeing. A higher weight increase could indicate that larger materials were absorbed on the paper surfaces. The P-treated paper specimens exhibited the highest weight change. According to the main components of different plant dyes, all of the rutin, curcumin, crocin and crocetin molecules included hydroxyl structures, which can form hydrogen bonds with paper cellulose and semicellulose and may be contribute to the weight increase. Other materials, such as solvent water, could be absorbed by these colorant dyes. The molecular weight and the number of hydroxyl structures in the dyes affected the weight change of the paper. For these different dyes, berberine has no hydroxyl structure; however, the presence of nitrogen means it could be positively charged with the ability to form a salt. Rutin has the largest number of hydroxyl groups and highest molecular weight of the colorant materials, which might be why the P-dyed paper exhibited the highest weight increase. These absorbed materials might act as barriers to defy attack from the paper's environment.

To further identify the effects of different colorant components on paper, TG analysis was used to obtain the mass loss as a function of temperature for different samples. Figure 8 shows that the weight loss for all samples was between 280 and 400 ℃, which corresponds with paper fiber decomposition, resulting in volatiles and low molecular weight materials such as CO_2, CO, ketones and aldehydes. For the nondyed paper, the removal of free water began ca. 55 ℃. The highest decomposition temperature was ca. 356 ℃. After that, the weight loss of the samples was small, and a carbonization process and rearrangement of aromatic rings began. Figure 8 shows that the A dyed paper specimens had higher decomposition temperatures than the undyed paper. However, the G-, T- and P-dyed papers displayed lower onset and end decomposition temperatures. The different TG results demonstrated that there were chemical bonds between the colorant and paper fibers. The chemical bonds could affect the thermal stability of the paper samples. According to the characteristic components of different dyes, the decomposition of rutin, crocin and curcumin could produce weak acid materials due to the existence of carboxyl and ketone groups, which could accelerate the decomposition of paper fibers under high temperature conditions. The experiments demonstrated that the dyed papers had lower thermal stability. The G-dyed paper had a higher weight increase and decomposition temperature than the P- and T-dyed papers. This result meant that the hydrogen bond between the paper and colorant dyes could affect the decomposition temperature. The A dyed paper had a higher decomposition temperature than the nondyed paper, which could be ascribed to the fact that berberine more strongly interacted with the paper fibers. The FTIR results (Fig. 9) showed a chemical bond ca. 1337 cm^{-1}, which was ascribed to the aromatic amine of the A dyed paper, while the other yellow dyed papers had no such characteristic bond. The aromatic amine of A could form a chemical bond with the hydroxyl groups of the paper fibers through nitrogen. The TG results further indicated that the positively charged nitrogen and hydroxyl groups exhibited a stronger interaction than the hydrogen bonds of the oxygen and hydroxyl groups.

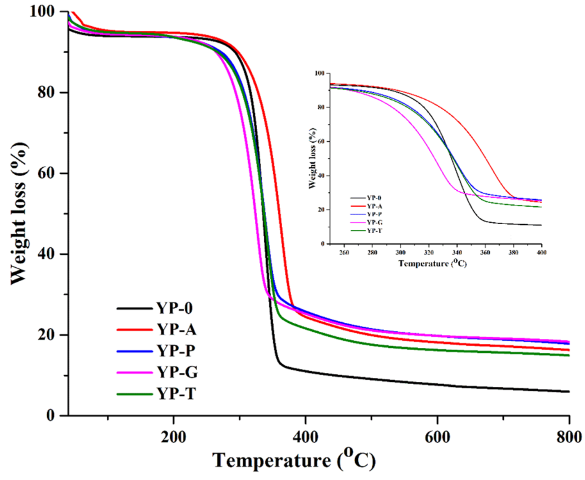

Fig. 8 TG results of dyed papers

To identify how the toning materials affected the mechanical properties of paper, tensile strength and folding endurance experiments were conducted. The mechanical strength of handmade paper is determined by the intrinsic strength of the fibers and the bonding strength between fibers. Additionally, the tensile strength of handmade paper is different in different directions. The tensile strength of traditional handmade paper usually depends on the orientation along the longitudinal direction (LD) and transverse direction (TD). The average values of the tensile strength and breaking length are shown in Figs. 10 and 11. The undyed paper had tensile strengths of ca. 21.8 N along the transverse direction and 19.6 N along the longitudinal direction. Slightly better tensile strengths and folding endurance were observed for the P-, T- and G-dyed papers. However, the A-treated paper specimens led to a distinctive improvement in the tensile strength and breaking length. These results could be explained by the different characteristic components of these colorant dyes, which affected the intrinsic resistance of the paper fibers within the experimental accuracy. The tensile strength is related to the binding force between fibers. The increases in the tensile strength and folding endurance were attributed to the formation of more electrostatic interactions due to the chemical reaction between the colorants and paper fibers. Furthermore, the A dye contained sugar materials that covered the fiber surface and led to an increase in the mechanical strength. To explain the mechanical properties, the surface paper specimens were further observed by SEM.

According to our Herzberg dye testing [28, 29], the main plants in the handmade paper specimens were Sinocalamus bamboo (*Sinocalamus affinis* (Rendle) McClure) and mulberry bark (*Morus alba* L.). SEM observations (in Fig. 12) indicated that the main plant included round tubular mulberry bark; the surface of the sample was uneven, and there were a few holes in the longitudinal direction and transverse stripes in the horizontal direction [30]. Joints, ravines and grooves were seen in the bamboo fibers [31]. After dyeing, the fibers were deposited by toning particle materials, some of which filled into the cracks, holes and ravines of the fibers, leading to a reduction in the porosity and pore size distribution. The filled colorant dyes could form chemical bonds with fibers by hydrogen bonds. This might be the reason for the improved mechanical

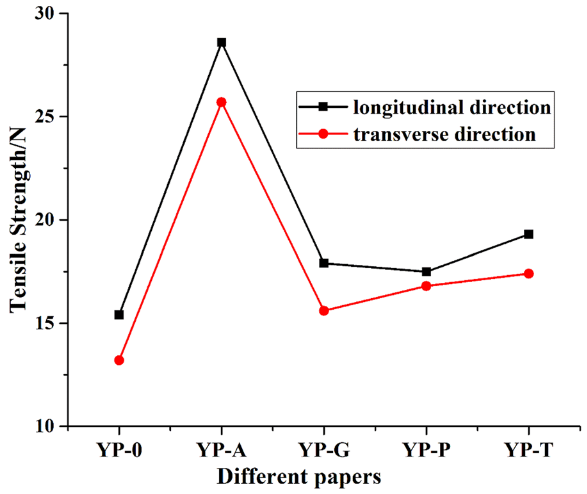

Fig. 10 Tensile strength values of dyed papers

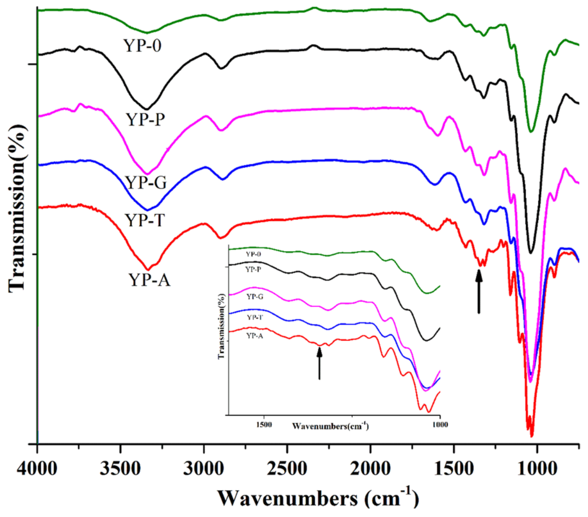

Fig. 9 FTIR spectra of dyed papers

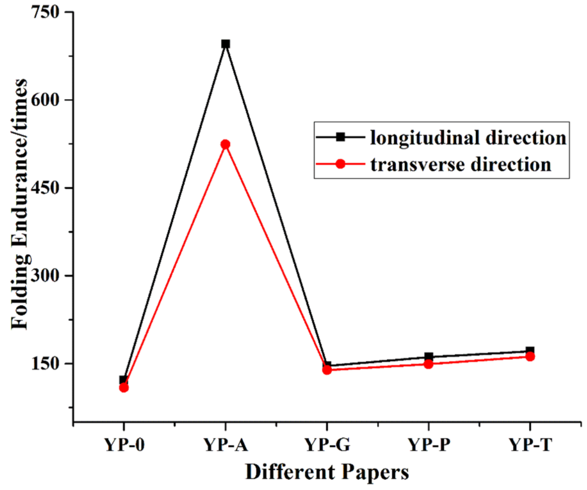

Fig. 11 Folding endurance values of dyed papers

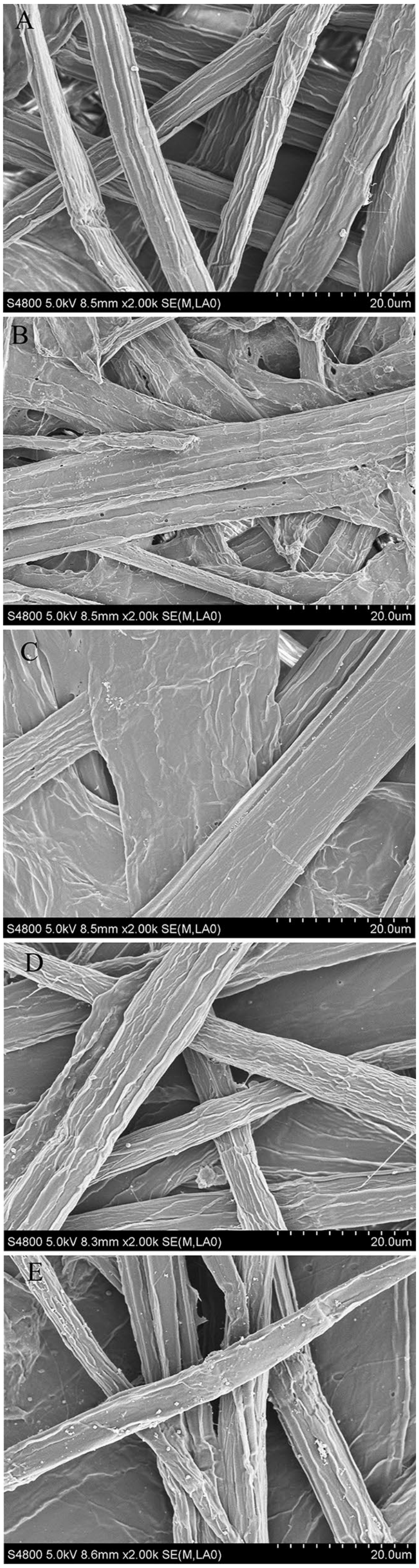

Fig. 12 SEM images of different papers. **A** undyed paper, **B** Amur cork tree dyed paper, **C** pagoda bud dyed paper, **D** gardenia dyed paper, E. turmeric dyed paper

properties of all the dyed papers. The best mechanical properties found, induced by A, were ascribed to the higher interfacial interaction due to the network and viscous substance coating on the paper surface and connecting the paper fibers. The mechanical test results confirmed that the electrovalent chemical bonds of the nitrogen and hydroxyl groups between the fibers and A were stronger than the interactions of the hydroxyl bonds induced only by the oxygen and hydroxyl groups. We will discuss how A affects the properties of the paper further in future work.

Conclusions

This experiment confirmed that handmade Daqian paper dyed with traditional botanical dye sources, such as Amur cork tree, turmeric, pagoda bud and gardenia, could have different properties. The different botanical sources had different effects on the properties, including the color, thermal stability and mechanical properties, due to their main characteristic components. The improved tensile strengths and folding endurances of these toning materials after dyeing are beneficial for paper. The chemical interaction between the Amur cork tree dyes and paper fibers had a stronger effect on the mechanical and thermal stability than those of the gardenia-, turmeric- and pagoda bud-treated paper due to the stronger hydroxyl-nitrogen bond and hydroxyl groups and viscous substances. This stability may be one of the reasons why most of the ancient books that currently still exist were dyed with yellow Amur cork tree dyes. All of these chosen yellow plant dyes slightly decreased the pH of handmade paper. We should be cautious when choosing how to conserve paper artifacts because some of them have a low pH, which might affect the conservation of the paper artifacts.

Abbreviations

TG: Thermogravimetric; LD: Longitudinal direction; TD: Transverse direction; ATR-FTIR: Attenuated total reflection flourier transformed infrared spectroscopy; SEM: Scanning electron microscopy; CIE: International Commission on Illumination; YP-0: The undyed paper samples; YP-A, YP-P, YP-G, YP-T, the Amur cork tree (A), pagoda bud (P), gardenia (G) and turmeric (T) dyed paper samples, respectively.

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Authors' contributions

Data were collected by YBL, XJZ. YBL and XJZ prepared and 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
Not applicable for that statement.

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