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The effect of traditional amur cork tree bark extract dyes on thermal stability of paper by accelerating ageing

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Abstract
Extracts from amur cork tree barks contain some of the most important traditional natural dyes used, especially for functional production in ancient Chinese papers, due to religious reasons and their aesthetic aspects and antibacterial properties. Some ancient paper artefacts, dyed with this colourant and scattered around the world, are currently preserved to different extents. This study aims to characterize the thermal degradation process of amur cork tree bark extracts dyed papers to improve the knowledge and effectiveness of restoration practices. The properties of extracts dyed papers treated under dry-heat accelerating ageing conditions were investigated via optical observation and scanning electron microscopy as well as with pH, tensile strength and folding endurance analyses. The results showed that the main components from extracted dyes played important roles in affecting the properties of dyed paper. The changes in surface colour, pH, morphology and mechanical properties after the artificial dry-heat ageing tests revealed the most suitable concentration of extract dyes for maintaining the long-term thermal stability of the paper.

Keywords: Dry-heat, Artificial ageing, Amur cork tree bark extract, *Daqian* paper

Introduction

Dyeing Paper with natural yellow plant dyes is one of the most important traditions in ancient China due to religious reasons, the high tinctorial strength of plant dyes, the vividness of the yellow colour, the insecticidal properties of yellow alkaloid dyes, and conservation considerations. Dyeing papers are also commonly used to dye matching papers when restoring cultural paper relics and maintaining old features. The analytical characterization of natural yellow dyes, including identifying the main components and investigating sources of natural yellow dyes in ancient artefacts, has attracted the attention of many researchers [1–8]. Sources of protoberberine alkaloids, as one of the most important natural yellow dyes, have been widely studied [8–12]. Components in the extract liquor include protoberberine components, viscosity materials and some weak acids. However, the amounts and ratios of protoberberine components vary as a function of the growth environment of the plant and based on which parts of the tree are sampled; berberine always predominates in *Phellodendron amurense Rupr.* (the inner part of the bark is called amur cork tree bark) [9]. In China, protoberberine is usually produced from two kinds of cork trees, *Phellodendron chinense* and *Phellodendron amurense Rupr*. According to our previous study, amur cork tree bark extracts have a better effect than that of other botanical-sourced yellow dyes in improving the mechanical properties of paper [11]. The dyeing mechanism of amur cork tree bark extracts is due to its major colour component, berberine, which is a cationic alkaloid that can be bonded to paper fibers by hydrogen bonds and electrostatic attractive forces [12]. Nevertheless, as a natural dye, colourant dyes from amur cork tree barks have some disadvantages; specifically, these dyes can fade and easily deteriorate due to heat,

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acid gases, light, etc. To date, the fading of natural dyes by light has been widely studied [13–16]. Indeed, valuable ancient books and other paper materials dyed with extracts from amur cork tree barks usually change colour considerably, even under no light conditions. Therefore, the need for protection against the damaging effects of environmental conditions is a very important concern for indoor conservation in the long run for amur cork tree bark extracts dyed paper. However, less attention has been paid to environmental factors, such as heat-induced degradation.

Considering all of the abovementioned issues, a laboratory design that can adequately simulate heat-induced degradation conditions under a controlled environment such as elevated temperature over a period of hours, is needed to provide information concerning the ageing of natural paper that may occur over a period of time. Currently, the thermal stability of amur cork tree bark extracts dyed paper is not well understood. In this study, colourant extracts from amur cork tree barks were prepared, and handmade *Daqian* paper was dyed without the use of a mordant. The dyed paper samples with different concentrations of extracts from amur cork tree bark were aged under artificial dry-heat ageing conditions. Our intent in this research was to improve the knowledge of the restoration of traditional amur cork tree bark extracts dyed paper by examining the effect of dyes on the thermal stability of paper under accelerated ageing conditions.

Experimental section

Materials

Daqian handmade paper (P) was supplied by the *Daqian* Paper Shop in Sichuan Province and had a grammage of 25.46 g/m^2 and thickness of 94 μm. Its main plants were Sinocalamus bamboo (*Sinocalamus affinis* (Rendle) McClure) and mulberry bark (*Morus alba* L.) based on Herzberg-dye testing. *Daqian* paper was made according to the traditional process of Chinese handmaking paper without fillers or sizing. The paper exhibited a weak alkalinity (pH = 7.8) due to the application of lime to remove impurities by retting and steaming natural plants to obtain white fibers.

Dried amur cork trees barks were purchased from a traditional herbal market in Chengdu, Sichuan Province. All materials were purchased commercially and used as received.

Preparation of colourant dyes

Detailed information on the preparation of amur cork tree bark extracts-dyed paper was described in our previous paper [11]. The dyeing process was based on traditional Chinese recipes adopted for laboratory procedures [17, 18]. Amur cork trees barks (50 g) were cut into slices and soaked thoroughly in 500 mL of deionized water for a period of 12 h at room temperature, followed by boiling and simmering for 30 min. The extracted solution was filtered to obtain high-concentration extracts from the boiled dregs using a cloth sack (the mass ratio of dissolved matter was ca. 6 ~ 8 wt%). The original extract liquor that was obtained from the amur cork tree bark was labeled as A0 and then was used for dyeing at certain liquor ratios.

Sample preparation

Liquors with different concentrations of amur cork tree bark extracts were obtained by diluting the original amur cork tree bark extracts (A0) with deionized water. The amur cork tree bark-dyed paper was prepared by completely immerging original undyed papers in colorant liquor at 80 ~ 90 °C and subsequently pulling the papers out of extract dyes slowly. Then, the dyed paper was hung on glass rods at 23 ± 1 °C for 48 h [11]. The paper samples at water/extract liquor ratios of 0:1, 1:1, 2:1 5:1, and 10:1 (*v/v*) were prepared and labelled as P-A0, P-A1, P-A2, P-A5 and P-A10, respectively. Photographic images of the colourant dyed papers are presented in Fig. 1.

All the paper samples were conditioned according to the International Organization for Standardization (ISO) 187–1990 [19] before mechanical measurements at a temperature of 23 ± 1 °C and a relative humidity (RH) of 50% ± 2% for 24 h. Tensile strength and folding endurance test samples were prepared. Samples that were 15 mm wide (with sides within 0.1 mm) and 250 mm long were used for tensile strength tests, and samples that were 140 mm long were utilized for folding endurance

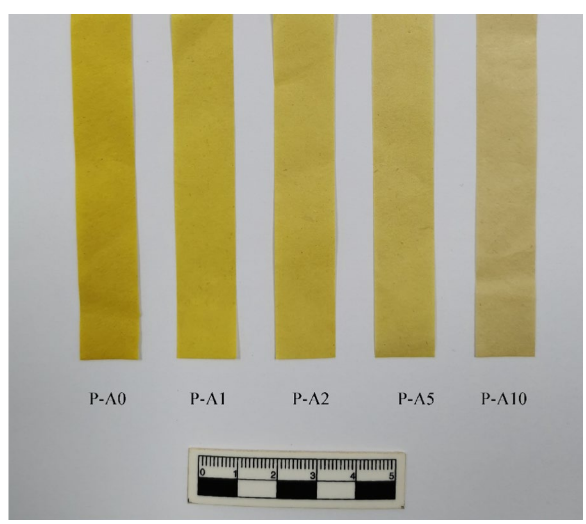

Fig. 1 Samples before aging

tests in the horizontal and transverse directions. It was ensured that the paper strips were free of abnormalities, wrinkles and creases.

Thermal stability studies

Samples were preconditioned according to ISO 187–1990 [19] (23 ± 1 °C and 50 ± 2% RH) for 24 h and cut into suitable sizes for testing. The thermal degradation temperature was chosen based on ISO 5630-1-1991 (Paper and board-accelerated ageing- Part 1: dry heat treatment at 105 °C) using an accelerating dry-heat ageing test [20]. The ageing test was performed in a constant climate chamber KMF 240 (Binder GmbH, Germany) at a temperature of 105 ± 1 °C for 80 days. At predetermined periods, specimens for each test were removed from the oven and conditioned for 24 h.

Characterization

pH tests

The pH of the colourant dyes was tested by a portable pH measurement device (Horiba, LAQUA twin-pH-22). Three to four drops of colourant dyes were allowed to rest on the flat sensor until the measured value was displayed.

The pH values of the paper samples were measured by cold extraction according to ISO 6588-1:2020 [Paper, board and pulps-Determination of pH of aqueous extracts-Part 1: cold extraction] [21]. The samples were cut with a cutter into pieces approximately 1 cm^2 in size. Second, 2 g ± 0.1 g air dry sample was extracted in 100 ml of cold distilled water for an hour. Then, the extract was filtered and 2 ml of 1 M KCl solution was added. Finally, the pH value of the cold extract was analyzed using a Mettler Toledo S400-B SevenExcellence™ pH meter (Mettler Toledo, Switzerland) at a temperature between 20 and 25 °C. The pH was recorded with 30 s when there was no measurement drift. The reported values were the mean of five duplicate determinations.

Colourimetric measurement

A solid reflection spectrophotometer (CM-700D from Minolta Co., Japan) was used to measure the colour changes caused by the effect of accelerated dry-heat ageing according to the standard ISO 11475:2004 [23]. The measurements were performed by a 10° observer and a D_{65} light source. The CIE L*a*b* colour space was used to determine the colour difference (ΔE) by the equation $\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$, where Δa is the red/green difference, Δb is the yellow/blue difference, and ΔL is the lightness difference. The average data and standard errors of ten measurements were calculated.

Mechanical property measurements

The mechanical property test samples were conditioned according to ISO 187-1990 [19] before testing. Tensile strength tests were performed according to the Technical Association of the Pulp and Paper Industry (TAPPI) T-494 and ISO 1924 standards [24, 25] with a TMI 84-56 tensile tester (horizontal) (Testing Machines, Inc., Holland) at a temperature of 23±1 °C, RH of 50 ± 2% and a test speed of 25 mm/min using an extensometer gauge of 180 mm × 15 mm. The reported values were the mean from ten determinations.

The folding endurance experiments were performed on a TMI 31-23 double folding endurance tester (Testing Machines, Inc., USA) according to TAPPI/T511 [26] and ISO 5626:1993 [27]. The applied force was 0.5 kg, and the double-fold force was 175 per minute. The folding endurance tests were carried out in a standard atmosphere (at a temperature of 23 ± 1 °C and an RH of 50% ± 2%).

Softness tests were performed on a softness tester (ZB-RR1000, China) at room temperature with a slit width of 5 mm according to the standard GB/T8942-2016 [22]. The paper samples were prepared by cutting samples that were 100 mm wide and 100 mm long in the horizontal and transverse directions, respectively. The reported values were the mean from ten determinations.

Microscope examination

Scanning electron microscopy (SEM) images were recorded using a Hitachi S-4800 instrument that was operated at a 5 kV working voltage. All specimens were sputter coated with gold prior to examination.

Degree of polymerization (DP) test

The degree of polymerization (DP) was determined from the measured change in the intrinsic viscosity; a fast and convenient method to estimate the average DP of cellulose is to use the empirical Mark-Houwink equation [28, 29].

$$[\eta] = 0.91DP^{0.85} \tag{1}$$

where, [η]: intrinsic viscosity, mL/g

Intrinsic viscosity [η] was measured using an Ubbelohde viscometer using cupriethylenediamine (CED) as the solvent at 25 ± 0.1 °C according to standard GB/T37838-2019 (Pulps-Determination of limiting viscosity number in cupriethylenediamine solution) [30]. CED was supplied by the China National Pulp and Paper Research Institute. The proportion of dry matter in the paper was determined according to GB/T 426-2008 (Paper, board and pulp- Determination of moisture content of analytical sample) [31]. The detailed procedure was described in our previous paper [32].

Results and discussion

The temporal appearance of the dyed paper with different concentrations of amur cork tree bark extracts is shown in Fig. 1. The paper that was dyed with undiluted amur cork tree bark extracts had a bright yellow colour. The colour became lighter with decreasing concentrations of dyes. The effect of colour changes on the paper samples under dry-heat ageing is shown in Fig. 2. Ageing could affect paper colour changes with different extract concentrations. The longer the ageing time was, the greater the colour change was. The initial bright yellow colour for P-A0 that was observed before ageing became dark brown after undergoing dry-heat ageing for 50 days. The colour difference values (△E) could indicate that the influence of ageing was more significant. The △E values of all samples are shown in Fig. 3. With dry-heat ageing time, there were some colour changes in the undyed paper after 50 days, although these changes were not significant (△E = 1.9). On the other hand, the paper dyed with undiluted amur cork tree bark extracts became brownish after only 5 days. △E increased quickly with dry-heat ageing times for the amur cork tree bark extracs dyed papers. The higher the concentrations of amur cork tree bark extracts were, the greater the colour change was. In this experiment, all of the dyed paper samples changed to brown to various degrees; that was, the L* and b* values decreased and the a* value increased with increasing concentrations of extract dyes. Researchers found that yellow berberine slowly turned under heating to deep red–brown–black crude products, including berberrurine [33–36]. The colour of the P-A0 sample changed to a slightly red–brown–black colour after dry-heat ageing was performed for 50 days, which meant that berberine decomposed to some degree during this ageing time. With decreasing concentrations of amur cork tree bark extracts, the colour did not change much, which meant that the main colour component did not decompose easily when the concentration of amur cork tree bark extracts was not as high. These results correspond with the description that when dyeing paper with amur

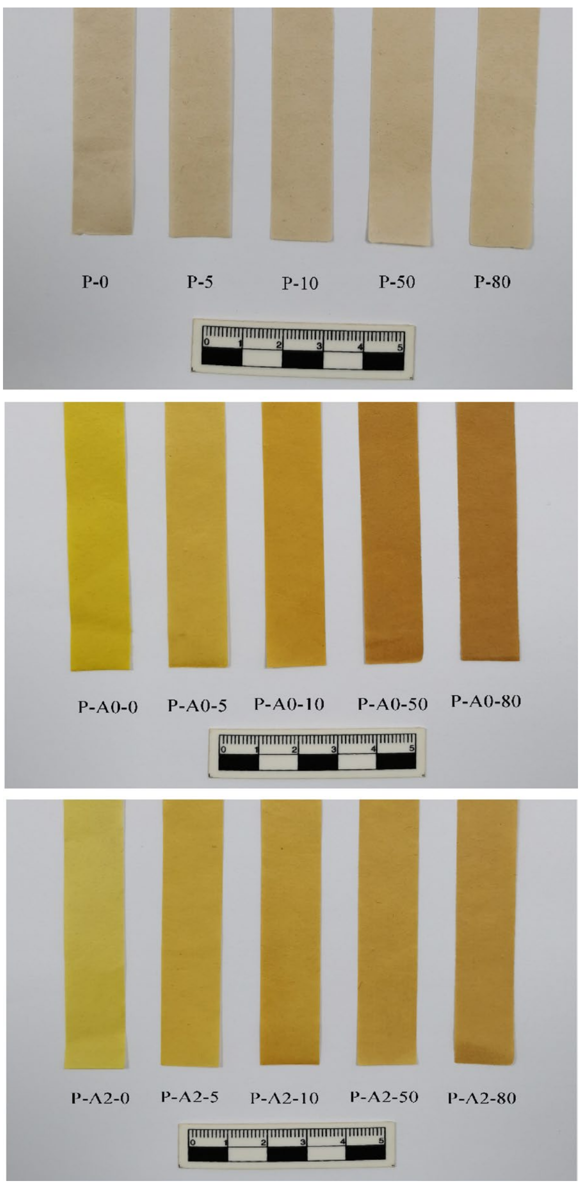

Fig. 2 Aged samples P, P-A0 and P-A2 at different aging times. The numbers from 0 to 80 represent ageing days

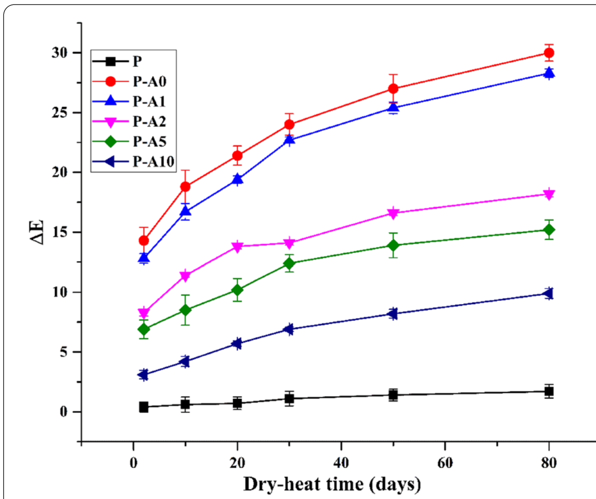

Fig. 3 Changes in color differences with dry-heat ageing times for different samples

cork tree bark extracts, an excessive amount of colorant should not be added or the colour will darken after a long period of time [37].

Colourant fading is associated with changes in characteristics, such as pH and mechanical properties. Mechanical property is an important direct indication of polymer properties. The mechanical properties of handmade paper are different in terms of the orientation along the transverse direction (TD) and longitudinal direction (LD). The paper's average values of tensile strength, folding endurance and softness with different extract concentrations were tested, and the results are shown in Fig. 4. The values of tensile strength, folding endurance and softness were affected by the concentrations of the amur cork tree bark extracts. The tensile strength and folding endurance of the original paper increased greatly after dyeing. As the concentration of the amur cork tree bark extracts decreased, the tensile strength and folding endurance of the dyed papers decreased gradually. However, even the tensile strength and folding endurance of P-A10 were both slightly higher than those of undyed paper. Compared with undyed paper, the dyed samples with A0 showed a slight increase in softness value which meant that paper became stiff after dyeing. However, the softness was not very different with decreasing concentrations of extract. As we know, the mechanical properties of dyed papers could be affected by the viscosities in extracts and chemical bonds between paper fibres and

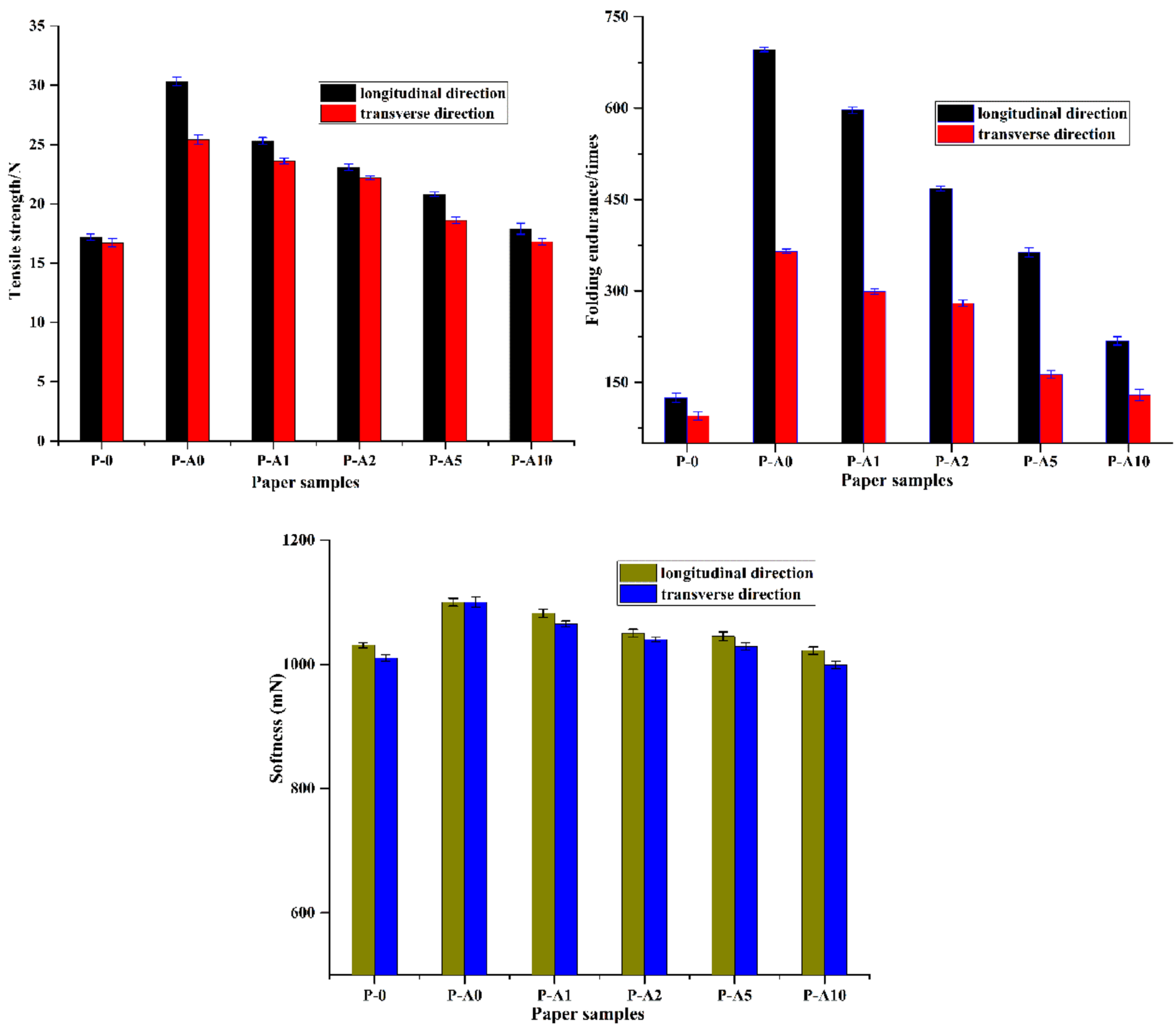

Fig. 4 Changes in mechanical properties before ageing for different samples

colorant dyes. We will discuss this further in the following sections. These mechanical results suggested that a suitable concentration of amur cork tree bark extract was important.

During ageing process, paper fibres can be depolymerized, which can lead to a loss in mechanical properties. In this study, two types of mechanical properties, tensile strength and folding endurance, were measured, and the results are shown in Figs. 5 and 6, respectively. The results showed that the mechanical properties of different samples decreased with increasing ageing time. Apparently, the patterns of the change for undyed paper and amur cork tree bark extracts dyed papers were similar, at least in the time dry-heat ageing range studied in this work. The tensile strength and folding endurance of P-A10 were both slightly higher than those of undyed paper under ageing periods. Although the thermal stability was not sufficient, samples with a water/extract ratio of 2:1 had higher thermal stability than that of other samples. It was very interesting that there was an increase in tensile strength, which began for P-A1 during the first 5 days, and this observation might be ascribed to the degradation of materials with low molecular weight and small and imperfect crystals under ageing conditions. Additional details are presented in the following section.

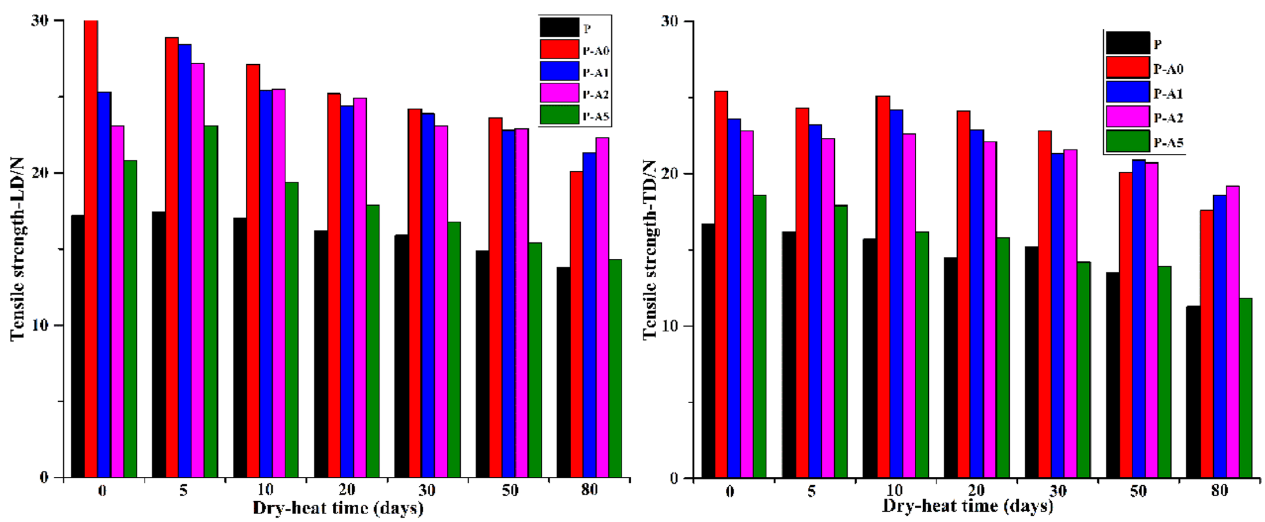

Fig. 5 Changes in tensile strengths with dry-heat ageing times for different samples

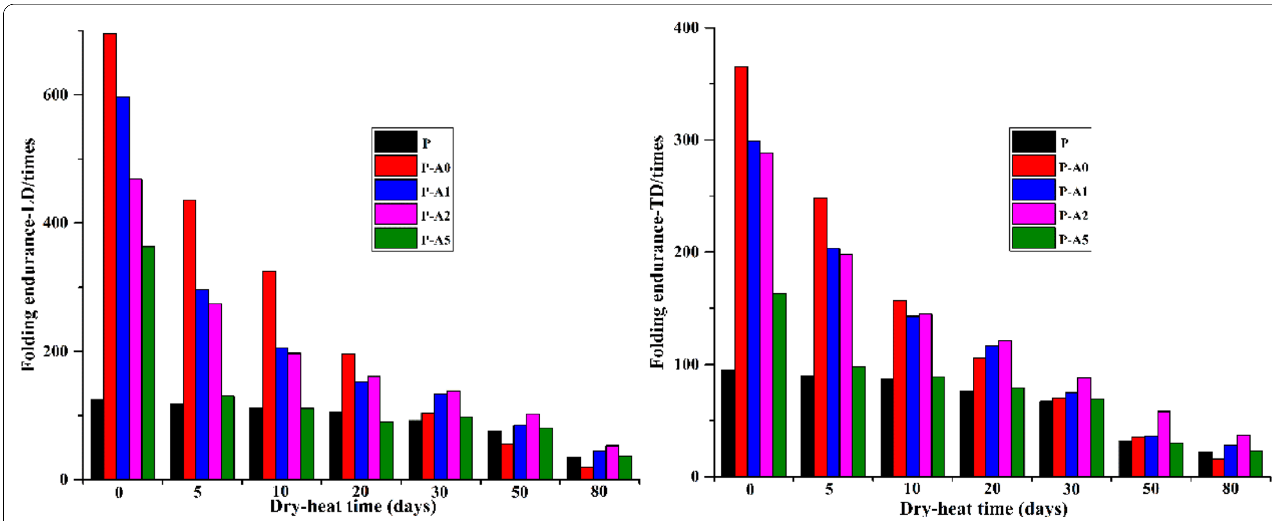

Fig. 6 Changes in folding endurances with dry-heat ageing times for different samples

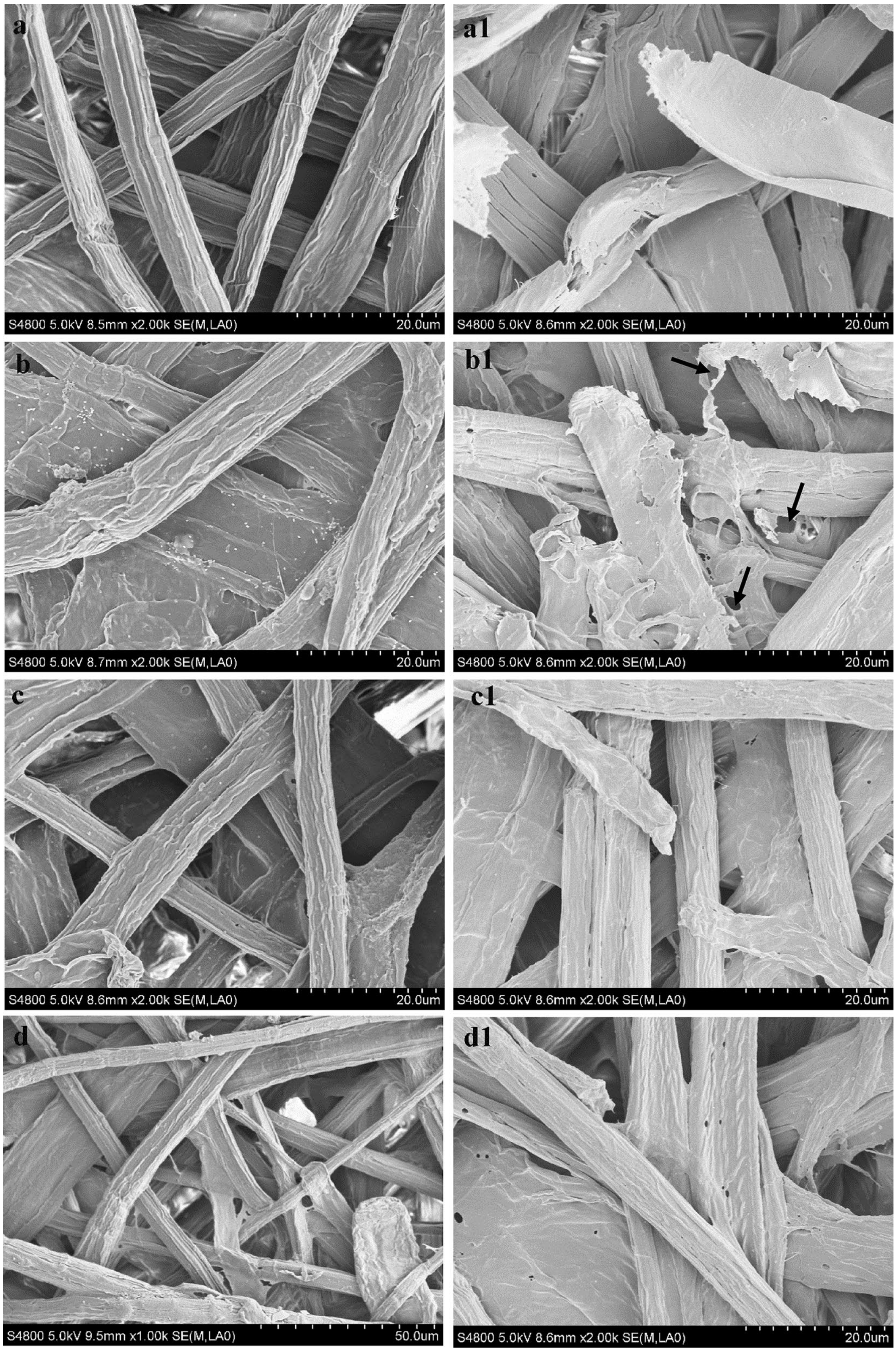

Fig. 7 SEM photography for the surface of P (a, a1), P-A0 (b, b1), P-A1 (c, c1) and P-A2 (d, d1) before (a, b, c, d: 0 day) and dry-heat 80 days (a1, b1, c1, d1: 80 days)

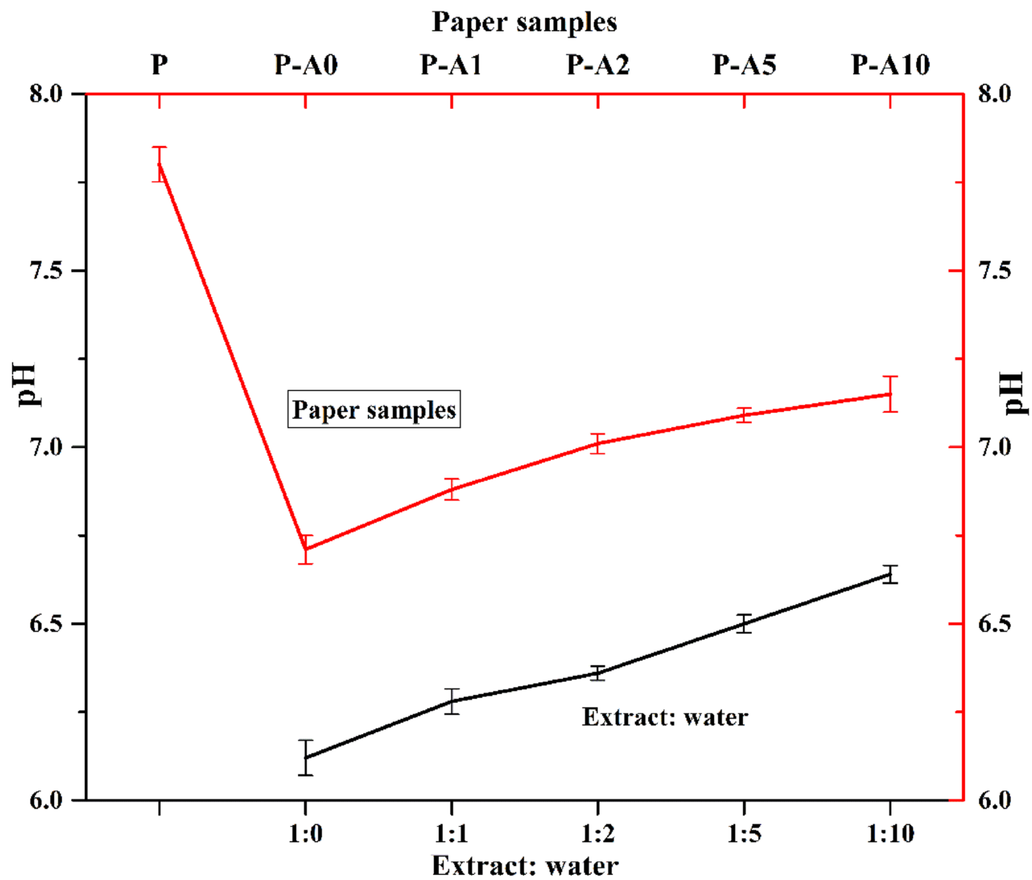

Fig. 8 Changes in pH for different paper samples and colour extracts

The morphologies of the samples during dry heating could be elucidated by SEM. Representative SEM images of the pure paper and dyed papers with different concentrations of amur cork tree bark extracts before and after being subjected to dry heat for 80 days are shown in Fig. 7. The pictures showed that some particle materials were deposited on the dyed paper fibres, as some components dissolved only in hot water. At the same time, agglomerations could be observed for P-A0 and P-A1, which would decrease the interaction between the main components and paper fibres. Figure 7 shows that all samples were destroyed to various degrees after thermal ageing for 80 days. Broken fibres were clearly observed on the surface of the undyed paper (Fig. 7 a1). The broken fibres and some holes indicated that fibres of P-A0 were destroyed more seriously after 80 days of ageing (Fig. 7 b1). Fewer broken fibres were observed with decreasing concentration of dye liquor, and only some small holes were observed on the surface of P-A2-80, which meant that P-A2 was not destroyed as much as the previous paper samples. The representative SEM images of the dyed samples revealed that lower concentrations of amur cork tree bark extracts exhibited a better dispersibility than that of higher extract concentration. A suitable concentration of extracts could help improve the stability of paper samples.

The paper's pH is considered one of the most important factors determining the paper's stability toward natural and accelerated ageing [27]. The pH of different concentrations of extract dyes showed that amur cork tree bark extract was weakly acidic (Fig. 8) due to existing acidic materials in the extracts, such as quinic acid and fatty acids [34]. Because the undyed paper (P) exhibited weak alkalinity (pH 7.8), papers that were dyed with extracts from amur cork tree barks exhibited a weak acidity to weak alkalinity, which should be ascribed to the residual alkaline materials in the paper during the papermaking process that neutralize the weak acidic materials in the extracts. Under dry-heat ageing conditions, the pH of all samples decreased (as shown in Fig. 9). According to Fig. 9, the decrease in pH for P-A0 was greater than that for other samples and resulted in a pH of 5.7 for P-A0. The lower pH of paper before ageing led to a lower pH after dry-heat ageing. These results showed that the

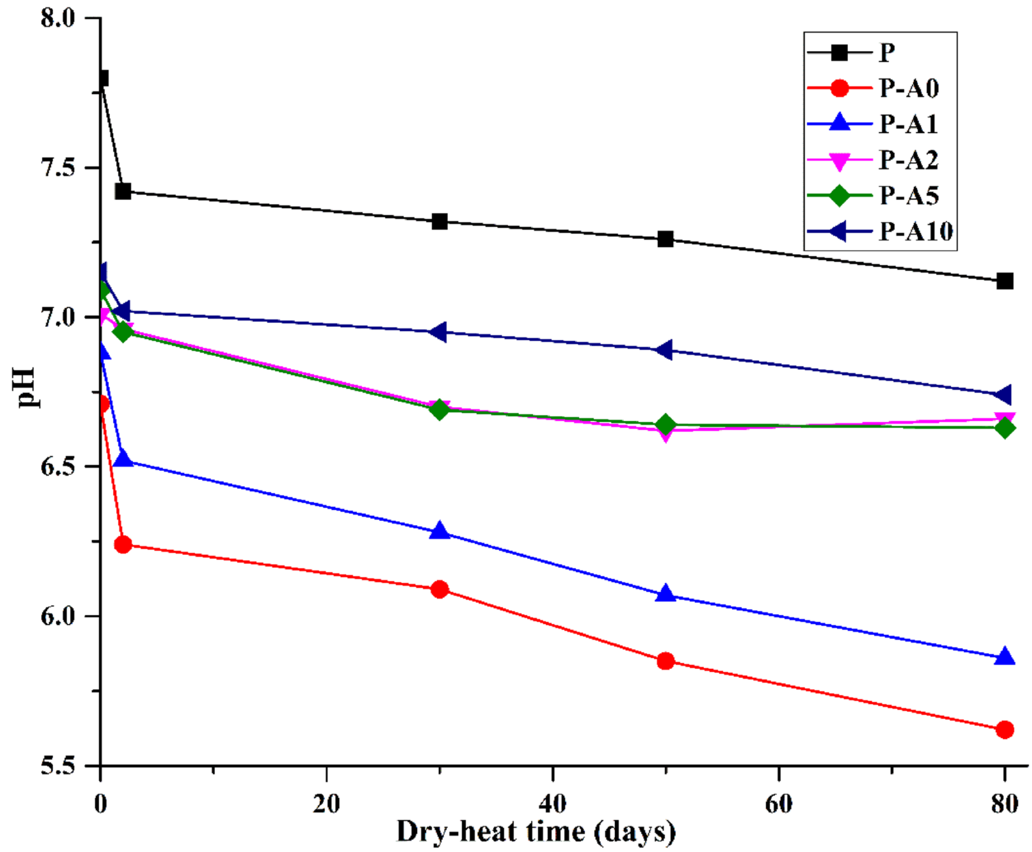

Fig. 9 Changes in pH with dry-heat ageing times for paper different samples

residual weakly acidic materials in dyed paper might accelerate the degradation of paper samples. Cheun reported that acidic materials might be produced during the ageing process for extracts from amur cork tree barks [34]. The accumulated components from degraded paper with components from the decomposition of amur cork tree bark extracts led to a decrease in pH. Furthermore, these weakly acidic materials accelerated the depolymerization of paper fibres and led to a decrease in the mechanical properties of A0 from the beginning of ageing.

The uniform dispersed extracts on the surface of paper fibres could decrease the degradation of paper by forming electrovalent bonds with paper fibres. This might be the reason why the pH of the samples at the A2 ratio did not clearly decrease. For P-A5 and P-A10 with fewer extracts, even though hydrogen bonds and other ionic bonds still existed between the paper fibres and extract liquor, these samples exhibited lower mechanical properties than that of A2 due to the fewer chemical bonds.

The ageing of paper under dry-heat conditions involves several main processes, namely, heat absorption, chemical bond cleavage and oligomer fragment formation. Therefore, the factors that affect the thermal ageing tendency of paper can control the degradation of paper. In the case of papers that are dyed with amur cork tree bark extracts, the uniform dispersed extract on the paper surface could improve paper's thermal stability by forming electrovalent bonds. At the same time, as a flavonoid compound, berberine directly removed some superoxide free radicals and some hydroxyl free radicals were formed by cellulose, hemicellulose and lignin during the ageing process of paper by means of single electron transfer, which might inhibit paper ageing to a certain extent. On the other hand, when the concentration of the extract liquor was higher, the existing weakly acidic residues were not neutralized, such as P-A0, which could accelerate the

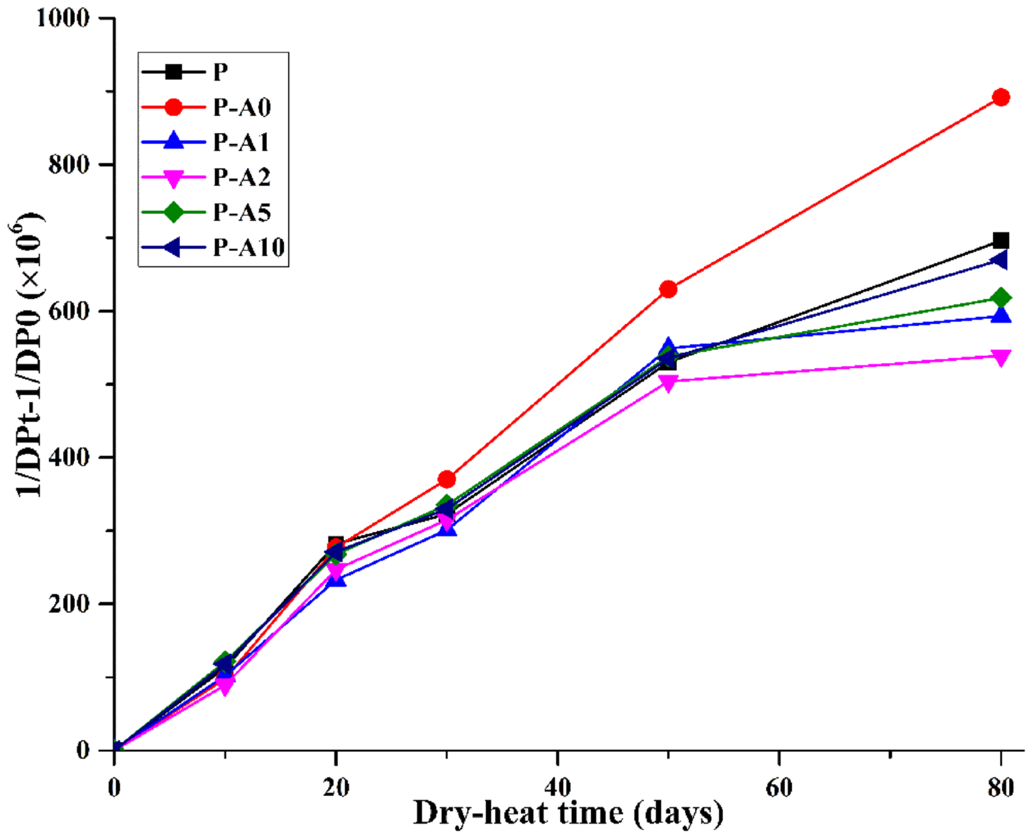

Fig. 10 Changes in DP with dry-heat ageing times for different samples

degradation of paper fibres. At the same time, agglomerated extracts might reduce the opportunity to form hydroxyl free radicals with cellulose, which facilitated the degradation of paper fibers.

The DP is a measure of chain length [32, 38]. The DP results of the paper samples were determined and are shown in Fig. 10. Interestingly, the DP of P-A2 and P-A5 was slightly higher than that of the undyed paper before ageing. However, the DP of P-A0 and P-A1 was slightly lower than that of undyed paper. This would be ascribed to the uniformly colorant dyes forming hydroxyl free radicals with paper fibres. These results further indicated that the dispersion of colourant materials on paper was very important to the properties of the dyed paper. The DP decreased when the paper samples were subjected to dry-heat ageing. The decrease in DP was more pronounced at higher concentrations of dye extracted from amur cork tree barks than at lower dye concentrations. This could be ascribed to the different pH values of the dyed papers. After dry-heat ageing was performed for 50 days, the DP of P-A0 was below half of the original values.

Conclusions

This study was an in-depth investigation of paper dyed with extracts from amur cork tree barks under dry-heat artificial ageing conditions. The handmade *Daqian* paper was dyed with amur cork tree bark extracts with different concentrations by diluting the original extract dyes (A0) with deionized water. The prepared samples were subjected to thermal degradation by accelerating the dry-heat ageing test at 105 °C for a period of time to provide information concerning the natural changes. Compositional and structural characteristics were determined by colour, pH, SEM, molecular weight, and mechanical property changes. Microscopic- and macroscopic- observations revealed that a high concentration of amur cork tree bark extracts induced agglomeration on the paper surface. The weakly acidic materials decreased the papers' pH and even led to weakly acidic P-A0. The residual acidic materials and viscosities in extracts affected the paper's properties. The residual weak acid, which could not be neutralized by alkaline materials in undyed paper, could accelerate the degradation of paper fibres. As an alkaloid compound, berberine can remove the free radicals formed during the paper ageing process by means of single electron transfer and the formation of chemical bonds between paper fibres and might inhibit paper ageing to a certain extent. High concentrations of extract liquors led to the agglomeration of colourant dyes and decreased the opportunity to form interactions between paper fibres. The present study showed that a water/extract liquor ratio of 2:1 (*v/v*) would be a suitable concentration for the preservation of amur cork tree bark extract-dyed paper.

In conclusion, traditional amur cork tree bark extract dyes affect the long-term thermal stability of dyed handmade paper. It is important to consider the concentration of colourant dyes when amur cork tree bark extracts are used to restore cultural paper relics.

Abbreviations

ISO: International Organization for Standardization; RH: Relative humidity; TAPPI: Technical association of the pulp and paper industry; LD: Longitudinal direction; TD: Transverse direction; SEM: Scanning electron microscopy; CIE: International Commission on Illumination; P: Undyed paper samples; A: Amur cork tree barks; P-A0, P-A1, P-A2, P-A5, P-A10: Indicated the water/extract liquor ratios of 0:1. 1:1, 2:1 5:1 and 10:1, respectively.

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Author contributions

Data of the research work were collected by YBL, YFW and TYW. QW and YBL prepared the manuscript. YBL revised it. 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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