A new reinforcement method for the conservation of fragile, double-sided, printed paper cultural relics

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Abstract

Paper cultural relics such as double-sided printed newspapers and periodicals represent modern cultural heritage. Severe damage such as embrittlement, decay, and dreg generally occur to paper; hence, it urgently needs conservation and restoration. Therefore, herein, a new reinforcement method for the fragile double-sided printed paper cultural relics is proposed with cotton mesh and adhesive PVA217 as reinforcement materials. Using a computer measured and controlled folding endurance tester, a pendulum tensile strength tester, pH meter, pulp viscosimeter, and other instruments, the physical properties of newspaper samples before and after aging were evaluated. In addition, the mechanical properties, chromatic aberration and surface morphology of simulated samples before and after the cotton mesh reinforcement and Japanese washi paper reinforcement were assessed. The experimental results indicate that this new reinforcement method could effectively improve the physical strength, chromatic aberration and durability of the simulated samples. Widespread applications of this cotton mesh reinforcement method for the reinforcement and protection of the red revolutionary cultural relics such as double-sided printed newspaper and periodicals during the Republic of China era are foreseeable.

Keywords: Double-sided printing, Paper cultural relics, Cotton mesh, Restoration and reinforcement, Japanese washi paper

Introduction

Since its invention, paper has become the primary medium for recording information throughout the world. Paper cultural relics (PCR) mainly include rare historical books, paintings and calligraphic works, newspapers, periodicals, etc., which are of great value in studies of culture, and history and ancient societies [1–5]. However, numerous paper cultural relics have become increasingly fragile though the ages in various environments and are in danger of severe damage [3–6]. The lignin oxidation [7] and acid-sizing [8] in these newspapers, periodicals, books and other double-sided printed paper cultural relics (DSPPCR) caused the paper fibers to hydrolyze and oxidize. In addition, environmental conditions including air pollution, temperature and humidity, pests, micro-organisms and UV light aggravate deterioration of PCR, in forms of yellowing, embrittlement, decay, breakage, dregs, and even breakage upon one single touch. As a result, the PCR are on the verge of self-integration [9, 10], as shown in Fig. 1. Hence, it is a challenge to effectively conserve and protect the double-sided printed-paper cultural relics due to the lack of scientific, reliable and practical technology for reinforcement and protection. Therefore, the research in reinforcement and protection of the DSPPCR in archives and museums is vital.

For protecting paper cultural relics, especially, those with printed words or characters on both sides, the ideal reinforcement materials should possess the properties of flexibility, transparency, long-term durability and
reversibility, etc. Previous researches on the paper reinforcement and restoration methods mainly include traditional mounting, silk mesh hot pressing, polyester film sealing, γ-radiation polymerization, electrospinning film, parylene crafting, and graft copolymerization coating [11–18]. However, all these methods have some unavoidable limitations. In the mounting method, it is hard to support the damaged paper with double-sided written or printed characters, and the mounting performance is not ideal. Silk mesh itself easily ages and falls off due to the aging-caused loss of adhesion. The polyester film changes the appearance, optical properties, texture, and thickness of paper, and easily hardens after aging. In the studies of effects of gamma irradiation on the paper relics, although gamma irradiation would affect the properties of paper, some researchers focused on the conditions in which the properties of the paper are affected as little as possible [19, 20]. Parylene protection needs to be performed under vacuum conditions, which involves complicated processes and strict conditions [21]. The graft polymerization coating can greatly protect the cellulose substrate, but the reinforcement is completed through chemical grafting reaction, which is difficult to separate after reinforcement, resulting in poor reversibility. Recently, one study [22] that caught our attention has introduced the silk mesh reinforcement and Japanese washi paper reinforcement for fragile, double-sided, printed paper cultural relics. The results indicate that Japanese washi paper reinforcement presented better mechanical properties and durability of treated simulated samples than silk mesh reinforcement. Therefore, it can be seen that researchers are always exploring new efficient, convenient, harmless, and long-term stable reinforcement methods for these DSPPCR.

Under such a background, taking into consideration the actual requirements for restoring and reinforcing paper cultural relics, it is very important that the reinforcement materials should be well compatible with the raw materials forming the paper cultural relics in terms of the physical and chemical properties. Pure cotton yarn mesh and paper are both plant fibers. With the cellulose content close to 100%, its expansion and contraction rate are consistent with those of paper; hence, they are highly compatible. Pure cotton fiber is composed of several fine fibers similar to the rich hairiness. The structure endows the fiber with large specific surface area, which can enhance the mutual adhesion between the fiber and the paper. As a water-soluble synthetic polymer, polyvinyl alcohol (PVA) is non-toxic, odorless and transparent, and has good chemical resistance and high mechanical properties [23–25]. Therefore, PVA has been one of the most popular synthetic polymers in paper adhesive materials and packaging applications [26]. In this research, a new reinforcement method is proposed with cotton mesh reinforcement material and water-soluble adhesive PVA217 for the conservation of fragile, double-sided, printed paper cultural relics. By studying the effects of accelerated aging methods such as hygrothermal aging, dry heat aging, and ultraviolet (UV) light aging on the appearance, potential of hydrogen (pH), degree of polymerization (DP), lignin content and mechanical properties of modern newspapers, simulated newspaper samples were successfully prepared. Meanwhile, paper performance and conservation process have been comparatively studied between cotton mesh reinforcement and Japanese washi paper reinforcement. This cotton mesh reinforcement method is expected to lay the theoretical foundation and provide technical support for the conservation and restoration work on the acidified and decayed paper cultural relics, especially the DSPPCR.

**Experimental section**

**Preparation of reinforcement material**

A single cotton filament with a diameter of 75 μm was selected and woven on a netting machine to form a
square grid with the side length of 1.5 mm. And 5.0 wt% PVA solution was sprayed on the grid using a spray gun and dried at room temperature. Then, it was cut from the netting frame to obtain the cotton mesh as reinforcement material.

**Accelerated aging experiment**

Dry heat, hygrothermal and UV-light accelerated aging methods were applied to prepare the simulated newspaper samples and evaluate the durability of the reinforcement methods. The dry heat accelerated aging test was performed for 21 days in a DHG-9245 A aging chamber at 105 °C according to ISO 5630-1:1991. The hygrothermal accelerated aging test was performed for 21 days in a DHG-9245 A aging chamber at 80 °C and 65% relative humidity (RH) as per GB/T 22,894−2008. The UV-light accelerated aging test was performed for 21 days in an ultraviolet lamp 60 W and the vertical distance was 5 cm.

**Evaluation of simulated samples**

To simulate the embrittlement situation of ancient DSP-PCR, three accelerated aging treatments were carried out on modern double-sided printed newspapers to prepare the simulated samples. 8% Alum aqueous solution was brushed on the samples before aging. After natural drying, the average pH of the samples was measured to be faintly acid. The cold extraction method was employed for pH testing according to the ISO 6588-1:2012. Viscometric determinations of the average DP were performed according to the standard viscometric methods (BS ISO 5351:2004), using a pulp viscosimeter (PTITV2000/AKV, Frank-PTI, Germany) and fresh CED solvent (China Pulp and Paper Research Institute Co. Ltd, Beijing, China). Lignin content test was performed with three parallel experiments to obtain the average value according to the Pulps-Determination of acid-insoluble lignin (GB/T 747-2003).

**Evaluation of reinforcement effect and durability**

Tensile strength, folding endurance, scanning electron microscope (SEM), and chromatic aberration tests were performed to evaluate the results of the conservation treatment. The folding endurance of the 150 × 15 mm² size specimens was measured with the YT-CTM tester according to ISO 5626:1993 method. The tensile strength of 270 × 15 mm size specimens was measured with a QT-1136PC universal material testing machine (Gaotai Testing Instrument Co., Ltd, Dongguan, China) as per ISO 1924-2:1994 method. The samples were cut according to the machine (MD) and cross directions (CD) of the paper. The SU3500 tungsten filament SEM was utilized to characterize the samples, spray the gold 80 s on the sample surface with an ion sputtering instrument (30 mA), and observe and analyze the sample surface under an acceleration voltage of 5 kV. The X-RiteVS-450 spectrophotometer was applied to test the chromatic aberration ∆E, which was evaluated by the CIE L*a*b* system [27].

**Reinforcement process**

**Japanese washi paper reinforcement process**

As shown in Fig. 2, simulated newspaper samples were reinforced with Japanese washi paper (2 g/m²). A 1922 newspaper was used to describe the conservation workflow. First, deionized water was lightly sprayed on the object to relax the paper (Fig. 2a). After that, the wheat starch adhesive was brushed on one side of the newspaper with a soft wool brush (Fig. 2b), which was followed by placing the Japanese washi paper onto the newspaper (Fig. 2c). The size of the Japanese washi paper was slightly larger than the size of the newspaper. The Japanese washi paper was gently brushed in order to flat surface and disperse adhesive (Fig. 2d). Mechanical paper was placed on the lined newspaper with brushing and pressure to absorb the excess water and bond tightly to the surface of the aged newspaper (Fig. 2e). Then, the same reinforcement procedure as above was used to reinforce the other side of aged newspaper (Fig. 2f–i). Finally, the lined...
newspaper was mounted on the Paper Wall or dried by machine until it was sufficiently dried. The aged newspaper after this treatment is shown in Fig. 2j.

**The cotton mesh reinforcement process**

As shown in Fig. 3, simulated newspapers samples were reinforced with the cotton mesh (300-yarn counts). A 1922 newspaper was used to describe the conservation workflow. The first step is to relax the paper with little deionized water (Fig. 3a). Then, cotton mesh was laid flatly on the paper to be reinforced, and the paper and the cotton mesh were kept flat (Fig. 3b). After that, a clean towel, steamed at a high temperature, was dipped in distilled water, wring dried until no water dripped, rolled into a roll and rolled on cotton mesh to completely adhere the cotton mesh and the newspaper (Fig. 3c). The soft magnetic strip was adsorbed around the newspaper, fixing it on the repair table and allowing it to be dried naturally (Fig. 3d).

**Results and discussion**

**Evaluation of simulated samples**

**Variations in appearance before and after aging**

To assess the merit and demerit of one treatment, some simulated samples instead of the precious 1922 newspapers would be required in order to study the mechanical and optical changes in treated newspapers. For accurate assessment results, it is important that simulated samples possess the similar properties of the 1922 newspapers as much as possible. Up to date, these double-sided printed papers made in the early 1922 (Fig. 1) had already lost their mechanical strength and appeared yellow after being exposed in various environments. Lignin oxidation in these double-sided printed papers produced chromophores, causing papers to turn yellow. Yellowing is one of the early signs of the aging and deterioration of paper. The accelerated aging process significantly accelerates the rate of lignin oxidation, leading to reduced mechanical strength as well as changes in appearance. Three accelerated aging treatments were carried out on 2020 newspapers to prepare simulated newspaper samples. These three accelerated aging methods include dry heat aging, hygrothermal aging and UV aging. Variation in appearance of these simulated newspaper samples was as a simple evaluation method. As shown in Fig. 4, these simulated newspaper samples after three accelerated aging treatments appeared yellow and brittle, which are similar to the 1922 newspaper.

**Variations in pH values before and after aging**

Paper cultural relics come into contact with acidic gases such as nitrogen dioxide and sulfur dioxide in the air, and then combine with water molecules in the air to form acids, which further change their pH values. Thereby, pH value could be another parameter to evaluate these simulated newspaper samples. To achieve the embrittlement and acidification of modern newspaper samples during the accelerated aging process, alum aqueous solution was applied to brush the surface of modern newspaper samples, further adjusting their pH values before aging to be weak acid. As shown in Fig. 5, newspaper samples before aging showed the pH value of 5.98. After three accelerated aging treatments, the pH values of the simulated newspaper samples increased in the order: hygrothermal aged < UV-light aged < dry heat aged. These results indicate that excessive humidity and high temperature in the hygrothermal aging process have a greater effect on the formation of paper acidity than UV light and high
temperature. The pH values of these three simulated samples were very close to 3.31, the pH value of 1922 newspapers. It means that the simulated newspaper samples possess similar pH values of 1922 newspapers.

**Variations in degree of polymerization before and after aging**

From a microscopic point of view, acidic hydrolysis leads to irreversible depolymerization of cellulose chains. As a classic analytical method with inexpensive and relatively easy to perform, viscometry was employed for the estimation of cellulose depolymerisation. The test results of DP are listed in Table 1. The unaged newspaper is a 2020 machine-made paper, whose DP value is 399. This DP value is in line with previous reports [8]. After three artificial accelerated aging treatments, the DP values of the simulated newspaper samples increased in the order: hygrothermal aged (48) < dry heat aged (71) < UV-light aged (88). Among these accelerated aging methods, hygrothermal aging had a greater effect on the DP of simulated samples, which is attributed to that high temperature and excessive humidity exacerbate the hydrolysis process, damage fibers and change the DP value. To accurately evaluate the reliability of simulated newspaper samples, a 1922 newspaper was used as a reference to compare its DP value with others in simulated newspaper samples. The results suggest that the simulated newspaper samples have similar DP values with 1922 newspaper.

**Variations in lignin content before and after aging**

Previous studies reported that lignin is susceptible to oxidation and further affects paper colour change [7]. In this work, the lignin contents in modern 2020 newspapers, simulated newspaper samples and ancient 1922 newspapers were measured, respectively. As shown in Fig. 6, the lignin content of 18.67 in 1922 newspapers is almost two times larger than that in 2020 newspapers, which are mainly due to the different fiber compositions. Compared with 1922 newspapers, the simulated newspaper samples showed smaller lignin contents, which are in line with the colour change after aging shown in Fig. 4. After dry heat and hygrothermal aging treatments, the lignin contents in the simulated newspaper samples are close to that in 2020 newspapers, which indicate that these aging methods have little effect on the lignin content. Compared with these two aging methods, the UV light aging dramatically decreases the lignin content, which is attributed to that UV light causes the degradation of lignin [28].

**Variations in mechanical properties before and after aging**

In this research, artificial dry heat aging, hydrothermal aging and UV light aging were utilized to simulate the natural aging process of simulated newspaper samples, and their tensile strength and folding endurance were then measured after the artificial aging. The aging results are presented in Fig. 7. After three kinds of accelerated aging, simulated newspaper samples significantly lose the tensile strength and folding endurance. The tensile strength and folding endurance in the machine direction (MD) are larger than...
those in the cross direction (CD). As shown in Fig. 7a, the folding endurance of the simulated samples in the MD after dry heat, hygrothermal and UV light aging has decreased by 34.61, 43.42, and 37.01%, respectively. The corresponding values in the CD turn into 53.73, 42.59 and 56.26%, respectively. It is obvious that the accelerated aging process has a larger effect on the folding endurance in the CD than that in the MD. However, it is different that the accelerated aging process has slightly bigger effect on the tensile strength in the MD than those in the CD (Fig. 7b). The tensile strength based upon different accelerated aging procedures could indirectly reflect the extent of oxidative and hydrolytic damage to cellulosic fibers in simulated samples.

Evaluation of reinforcement effect

Variations in mechanical properties before and after reinforcement

Folding endurance is the most sensitive indicator of changes in the paper structure and expresses the suitability of paper better than other mechanical properties [29, 30]. Tensile strength is an important indicator, which reflects the cellulosic structure of paper and the microscopic behavior [30]. Figure 8 shows the effect of cotton mesh reinforcement and Japanese washi reinforcement on the folding endurance and tensile strength of the simulated newspaper samples. Compared with untreated newspapers, folding endurance and tensile strength in the MD and CD increased considerably after these two reinforcement treatments, which confirm their beneficial
reinforcement effects. As shown in Fig. 8a, the folding endurance of the simulated samples treated with cotton mesh in the MD after dry heat, hygrothermal and UV light aging has increased by 53.80, 69.94, and 58.42%, respectively. The corresponding values in the CD turn into 143.18, 108.72 and 154.07%, respectively. Compared with Japanese washi reinforcement, the cotton mesh reinforcement is more advantageous in improving the folding endurance of simulated samples, which is possibly due to the excellent flexibility of the cotton mesh. As observed in Fig. 8b, these two treatments show a smaller increase on tensile strength than the increase on folding endurance, which is attributed that folding endurance is the most sensitive indicator of changes in the paper structure [29]. Overall, Japanese washi reinforcement is more helpful to increase the tensile strength of simulated samples than cotton mesh reinforcement. The above results indicate these two treatments would be complementary in improving the performance of folding endurance and tensile strength.

Variations in chromatic aberration before and after reinforcement

In the experiment, the $\Delta E$ values of simulated samples treated with cotton mesh and Japanese washi paper are 1.07 and 1.64, respectively. After cotton mesh reinforcement, the value of $\Delta E$ lower than 1.5 could be seen as undetectable by the human eye [27]. Although the $\Delta E$ value of the paper treated with Japanese washi paper is slightly bigger than 1.5, it might be optimized further by adjusting thickness of Japanese washi paper or changing the concentration of the repairing paste.

Figure 9 shows the values of visible light reflectance from the surface of the simulated newspaper samples before and after reinforcement. It can be seen that the reflectance values of the sample surface in the 400–700nm band remains unchanged before and after cotton mesh reinforcement and their curves basically coincide. It further confirms that the process of cotton mesh reinforcement shows no obvious colour change of the sample surface. However, Japanese washi paper reinforcement decreases the reflectance of the sample surface, which is primarily attributed to that the repairing paste formed a film to affect its reflectance.

Durability evaluation of reinforcement methods

Durability is a key indicator in evaluating whether one treatment is suitable for the paper relics. The durability of paper refers to the length of time it can maintain various physical and chemical properties. Generally, the most important factors in paper degradation include temperature, moisture and light radiation. Therefore, the simulated samples before and after reinforcement were treated for 21 days with dry heat, hygrothermal, and UV light aging to obtain reliable data for the durability evaluation of reinforcement methods.

Variations in microscopic morphology before and after aging

SEM micrographs could provide microscopic information to evaluate the durability of the cotton mesh reinforcement and Japanese washi reinforcement. As shown in Fig. 10a, e, paper fibers of simulated samples treated with the cotton mesh and Japanese washi paper were relatively dense and evenly dispersed. However, after 21 days of dry heat, hygrothermal, and UV light aging, small cracks appeared on the surface of simulated samples treated with Japanese washi paper (Fig. 10b–d). It is speculated that the repairing paste dried and formed into a film, then it was broken after aged and caused the surface to crack. Meanwhile, it is noticed that the simulated samples treated with the cotton mesh after aging showed no obvious cracks (Fig. 10f–h). This is mainly due to the fact that water-soluble adhesive PVA217 on the surface of cotton mesh did not form an overall film on the paper and maintained the original surface morphology of the paper. All these structural characteristics would improve the physical strength of paper archives and enhance their aging resistance.

Aging resistance of the reinforcement samples

The durability results are shown in Fig. 11. Compared with unreinforcement, cotton mesh reinforcement and Japanese washi paper reinforcement significantly improved the retention rates of folding endurance and tensile strength. The durability of the reinforcement samples is about 2–3 times higher than that of the unreinforced samples. As shown in Fig. 11a, the retention rate of the folding endurance after cotton mesh reinforcement is between 84 and 93%, while the...
retention rate of the folding endurance after the Japanese washi paper reinforcement is generally between 78 and 86%. This result shows cotton mesh reinforcement has better durability than Japanese washi paper reinforcement from the view of folding endurance. Figure 11b demonstrates that the accelerated aging methods affect the retention rate of the tensile strength for reinforcement samples. For dry heat aging, cotton mesh reinforcement displays better durability than Japanese washi paper reinforcement. However, the durability is reversed for these two reinforcement methods after UV light aging. It is speculated that the difference in durability is related to aging resistance ability of their reinforcement materials. Overall, these two reinforcement methods significantly improve the durability of simulated newspaper samples, and their levels of durability is a little different in terms of folding endurance and tensile strength.

Comparison of reinforcement methods
Japanese washi paper, a thin and transparent material, which reinforcement will not affect the reading of the DSPPCR, and has been widely used in Taiwan, Hong Kong, Singapore, Japan and other places. However, because Japanese washi paper needs to be imported from Japan, this reinforcement method has not been used extensively for the reinforcement and protection of Chinese DSPPCR. The cotton mesh reinforcement in this research employs cotton fiber and adhesive PVA217 as reinforcement materials, which are widely available at low cost.
From the perspective of the reinforcement process, the cotton mesh reinforcement is completed with simple adhesion and compaction, while Japanese washi paper reinforcement requires more procedures including brushing paste, paper mounting, absorbing excess water, and compaction. For these two reinforcement methods, the paper relic conservators have successfully combined deacidification and reinforcement process in the actual conservation of fragile, double-sided, printed paper cultural relics [22]. Meanwhile, these two reinforcement methods both get the advantages of good compatibility, good reversibility, and compliance with the principle of minimum intervention for paper relic conservation.

From the results of reinforcement effect and durability, cotton mesh reinforcement shows slightly better advantage in the improvement of mechanical property, chromatic aberration, durability performance than Japanese washi paper reinforcement with 2 g/m² Japanese tissue. Inevitably, these two reinforcement methods have their own limitations. For example, Japanese washi paper reinforcement is not appropriate for the conservation of water-soluble handwriting that easily diffuses when exposed to water. The cotton mesh reinforcement may cause slightly aesthetic defects caused by gridlines. Overall, the comparison results show that the new method would be another option for the conservation of fragile DSPPCR besides the Japanese washi paper method.

**Conclusions**

To meet the requirements of the reinforcement and restoration of Chinese double-sided printed paper cultural relics, herein, a new reinforcement method is proposed with pure cotton mesh and water-soluble adhesive PVA217. The reinforcement method not only meets the compatibility requirements in bonding with paper, but is also in line with the reprocessing principle of reinforced paper under trace water wetting. Through pH value alteration of newspapers and artificial accelerated aging, the simulated newspaper samples could achieve similar properties to the 1922 newspapers. The mechanical strength and aging resistance of the simulated newspaper samples treated with cotton mesh reinforcement and Japanese washi paper reinforcement were significantly improved. After complete evaluation of the effect of two reinforcement methods on the mechanical property, chromatic aberration and surface morphology of the simulated newspaper samples, cotton mesh reinforcement shows slightly better advantage in the improvement of mechanical property, chromatic aberration, durability performance than Japanese washi paper reinforcement with 2 g/m² Japanese tissue. Therefore, widespread practical applications of this cotton mesh reinforcement method for the conservation of damaged DSPPCR are foreseeable.

**Abbreviations**

PCR: Paper cultural relics; DSPPCR: Double-sided printed paper cultural relics; SEM: Scanning electron microscope; UV: Ultraviolet; pH: Potential of hydrogen; DP: Degree of polymerization; PVA: Polyvinyl alcohol; MD: Machine direction; CD: Cross direction.

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**Authors’ contributions**

JL performed the conservation process of double-sided paper cultural relics and written the manuscript. HX and JW carried out literature and examination. XC and JC assisted in sample testing and data analysis. JC was involved in the initial concept of the examination. ZJ and YL provided financial support for this study. All authors have read and agreed to the published version of the manuscript.

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**Availability of data and materials**

All data are available on request.

**Declarations**

**Competing interests**

The authors declare that they have no competing interests.

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