Use of Bacterial Cellulose in Degraded Paper Restoration: Effect on Visual Appearance of Printed Paper

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One of the most common methods used to reinforce damaged paper is to apply a lining, using Japanese paper (JP). The reinforcing material must consolidate the paper without modifying its visual appearance. The unique properties of bacterial cellulose (BC) suggest that it could be efficiently used to reinforce degraded paper documents. The changes in the visual appearances of the printed commercial papers lined with BC and JP were examined in this study. Four commercial papers, coated and uncoated, were printed with cyan, magenta, yellow, and black offset inks. The printed samples were lined with BC and JP sheets. Print density, gloss, and CIELab coordinates were tested in the lined and unlined samples before and after aging. Lining with JP notably affected the print density and CIELab coordinates. The lining with BC resulted in lower decrements in color intensity. The gloss values of samples lined with BC differed widely amongst the papers, whereas in papers reinforced with JP these values never exceeded 6%. Subjecting the samples to an aging process did not markedly modify the results except for the BC-lined samples, in which color differences increased.

Keywords: Bacterial cellulose; Japanese paper; Lining; Optical properties; Paper aging; Paper restoration; Printed paper

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INTRODUCTION

The disappearance of bibliographic and documentary heritage is one of the main problems faced by libraries and archives around the world. The passage of time causes changes in paper characteristics such as yellowing, loss of paper strength, etc. However, the most notable effect is the acid hydrolysis of the cellulosic and hemicellulosic components (Baty et al. 2010). Paper conservators have attempted to limit paper degradation by adding alkali to neutralise the acids generated and also to build an alkaline reserve that can buffer future acid degradation (Ahn et al. 2013). Nevertheless, this method does not restore the flexibility and strength of the damaged paper and in many cases additional reinforcement treatments are necessary (Arderlean et al. 2011).

Lining is the most common method used to restore damaged paper that consists of applying a reinforcing layer over the paper surface using an adhesive such as wheat starch. Sheets of Japanese paper (JP) are commonly used as reinforcing layers in lining. The JP paper originates from Japanese long fibre species, such as kozo, mitsumata, and gampi, which provide high resistance with a very low basis weight (Bansa and Ishii 1997). However, these papers are not entirely satisfactory for all documents requiring restoration, as JP sheets are not very homogeneous and the lining method considerably increases the thickness of restored papers.
It has recently been demonstrated that other cellulose structures, such as nanocelluloses, enhance the mechanical strength of paper products and provide them better barrier properties, even at very low percentages. This makes the nanocellulose and nanofibrillated cellulose very promising materials for paper reinforcing purposes (Ioelovich 2008; Li et al. 2015; Hubbe et al. 2017).

Bacterial cellulose (BC), a specific type of nanocellulose, is produced by certain bacterial species, *e.g.*, those of the genera Acetobacter, Agrobacterium, and Aerobacter (Shoda and Sugano 2005). Although BC is similar to plant cellulose, it is free of lignin, hemicelluloses, and pectins. Furthermore, BC is biocompatible and non-toxic. It can also be produced without intensive energy consumption.

Santos et al. (2013, 2015) showed that cellulose layers produced by *Gluconacetobacter saccharoflamentans* have promising properties for use as reinforcing layers of degraded documents, mainly because of their unique characteristics: high crystallinity index, low porosity, good mechanical properties, and stability over time. When BC sheets were used as reinforcing layers to line old books and model papers (Santos et al. 2016a,b), the results revealed that the strength properties were as good as the samples reinforced with JP. In some properties, BC-lined samples showed better values, greater decreases in paper wettability, with very low air permeability, which could work as a barrier against external agents.

An outstanding advantage of BC film is that when it was applied as a reinforcement layer it did not noticeably increase the paper thickness (7 to 10 microns), whereas when JP was applied, this parameter increased to approximately 35 microns. In relation to visual appearance, the BC-lined papers showed slightly greater changes than the JP-lined papers in the optical properties in unprinted areas, but the characters were more legible with BC reinforcing. When the lined papers were subjected to an aging process, there were no notable differences between the BC and JP reinforcing methods except yellowing, which was more noticeable in the BC-lined samples (Santos et al. 2016a,b).

In studies on the accelerated aging of paper when conducted on handmade sheets, a moderate loss of brightness (4% ISO) and considerable yellowing (12%) were observed (Ardelean et al. 2011). Similar studies on commercial papers (both coated and uncoated) reported yellowness indexes higher than 15% (Karlovits and Gregor-Svetec 2012) and a loss of brightness of approximately 10% to 22% (Havlínová et al. 2002a). Information about how aging affects printed areas is limited, although studies about the durability of offset ink exposed to accelerated aging have shown different behaviour for each color (Havlínová et al. 2002b; Karlovits and Gregor-Svetec 2011). However, available literature includes no studies about the effect of the reinforcing layer on the visual appearance of restored paper and color durability.

The most common parameters used to evaluate the optical properties and visual appearance are print density, CIELab color coordinates ($L^*, a^*, b^*$), and print gloss. Print density is defined as the percentage of light reflected from the substrate or the ink, and is related to the color intensity. The $L^*$, $a^*$, and $b^*$ coordinates designate all of the colors visible to the human eye. The coordinate $L^*$ represents the lightness of the color, the $a^*$ coordinate gives its position between red and green, and the $b^*$ coordinate indicates its position between yellow and blue. Gloss can be described as the relative ability of printed paper to reflect light at the mirror angle (Hubbe et al. 2008). Although these parameters change after artificial aging (Havlínová et al. 2002b), there is no information about how a film with such low porosity as BC can protect the optical properties of printed documents.
Lining damaged paper with BC sheets has proven to be an excellent alternative to JP lining to improve the strength and stability over time. Although it is necessary to determine how BC lining affects the visual appearance of the original document, particularly the printed areas. To validate BC lining it is also important to study how the BC layer affects each offset color print and determine whether the BC-lined areas with print are stable over time.

To achieve this objective, four commercial fine papers with different furnish compositions and finish grade were selected. All of the paper samples were printed with cyan, magenta, yellow, and black offset inks, additionally enabling the study of the influence of paper grade and ink color on changes in the optical properties of lined papers. Printed test pieces were lined with layers of BC and JP. Lining was performed following the traditional method using wheat starch as an adhesive. Changes in optical properties were determined on the lined and unlined samples, and before and after artificial aging.

**EXPERIMENTAL**

**Reinforcing Materials: Bacterial cellulose and Japanese paper**

Bacterial cellulose films used as reinforcing material in this study were acquired as previously described (Santos et al. 2016a). Briefly, *Gluconacetobacter saccharofermans* CECT 7291 obtained from the Spanish-type culture collection (CECT) was used to inoculate 100 mL of a modified Hestrin and Schramm (HS) medium (Fructose, 20 g/L; Yeast extract, 5 g/L; Corn steep liquor, 5 g/L; Disodium acid phosphate (Na₂HPO₄), 2.7 g/L; and Citric acid, 1.15 g/L) in 150-mm petri dishes. Samples were maintained at 30 °C under static conditions and collected after seven days of culturing. To purify BC, the samples were first washed with distilled water, treated with 1% NaOH at 90 °C for 60 min, then exhaustively washed with distilled water after filtration through a medium porosity filter paper in a Buchner funnel, and air-dried.

The JP used (Tengucho, Awagami Papers, Tokushima, Japan) is one of the most common materials used in restoration. It is un-sized paper made with bleached kozo fibres and has a basis weight of 9 g/m² and colorimetric coordinates of \( L^* = 88.5, a^* = 0.65, \) and \( b^* = 1.69 \).

**Paper Samples and Printing Process**

The four commercial printing papers employed in this study are described in Table 1. The selection criteria involved the furnish composition and the finish grade. The papers were characterised by basis weight (ISO 536 (2012)), gloss (ISO 8254-1 (2009)), CIELab coordinates (ISO 11664-4 (2008)), and optical density (ISO 2846-1 (2006)). The gloss was determined in a D48-7 glossmeter (Hunter Associates Laboratory Inc., Reston, VA, USA) using an angle of incidence of 75°. The \( L^* \), \( a^* \), and \( b^* \) coordinates and optical density were measured with an X-Rite 500 Spectro-densitometer (X-Rite Inc., Grand Rapids, MI, USA).

The coated papers, the glossy-finished (GC) and the matte-finished (MC), and the wood-free uncoated paper (WU) showed similar optical densities and CIELab coordinates. The super-calendered paper (SC) had the lowest brightness as expected, because it is made from mechanical pulp. The gloss values differed widely among the papers examined and was consistent with the different finish grades.
**Table 1. Description of Papers Assessed**

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
<th>Optical Density</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>Gloss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC</td>
<td>High weight coated paper manufactured from chemical pulp and gloss finished</td>
<td>0.09</td>
<td>92.4</td>
<td>0.7</td>
<td>-2.9</td>
<td>75.4</td>
</tr>
<tr>
<td>MC</td>
<td>High weight coated paper manufactured from chemical pulp and matte finished</td>
<td>0.10</td>
<td>91.5</td>
<td>1.0</td>
<td>-2.3</td>
<td>21.1</td>
</tr>
<tr>
<td>WU</td>
<td>Wood-free uncoated paper manufactured from chemical pulp and sized finished</td>
<td>0.10</td>
<td>90.3</td>
<td>1.1</td>
<td>-4.3</td>
<td>5.8</td>
</tr>
<tr>
<td>SC</td>
<td>Super-calendared paper manufactured from mechanical pulp. No surface coating.</td>
<td>0.29</td>
<td>81.3</td>
<td>-0.4</td>
<td>7.6</td>
<td>51.4</td>
</tr>
</tbody>
</table>

A global standard tester 2 (IGT Testing Systems, Amsterdam, Netherlands) and a rubber-coated printing disc were used to print the paper samples. The test conditions employed were 1.0 m/s printing speed and 400 N print force. All of the papers were printed with the four commercial offset inks (cyan, magenta, yellow, and black, provided by Arzubialde S. L, Logroño, Spain) at the print density targets of each color (1.45 ± 0.05 for cyan, 1.40 ± 0.05 for magenta, 1.35 ± 0.05 for yellow, and 1.70 ± 0.05 for black). The samples were always printed on the same side. The print density, CIELab coordinates, and gloss were measured 24 h after printing.

**Lining of Printed Paper and Aging Procedure**

In archival contexts, lining refers to the process of covering a sheet of archival paper with stronger materials to strengthen the paper. In this study, JP and BC sheets were used to line the printed samples, *via* wheat starch as adhesive. Samples of each color print were lined with BC in one part and JP in another, resulting in two fields with a different lining on each test piece. Both the reinforced and non-reinforced color printed samples were subjected to an accelerated aging process according to ISO 5630-3 (1996) at 80 ºC and 65% relative humidity for 144 h. An unprinted control test piece of each paper was also subjected to aging. A diagram of the treatment sequence is shown in Fig. 1.

![Fig. 1. Treatment sequence: aging (a), printing (p), and lining with BC or JP for GC paper](image)

The quality of the printed samples was measured after aging, lining with BC or JP, and further aging. Print density, CIELab coordinates, and gloss were measured as described in the previous section. These parameters were also tested in the unprinted samples after aging.

The color differences between lined and unlined samples were calculated. This index was obtained as a single value from the Euclidean distance between two points of the CIELab space that explained how closely the resulting color after the treatment process matched the original printed color. The color difference $\Delta E^*$ (SCAN-G 5:03) between two stimuli was defined as,

$$\Delta E^* = \sqrt{(L^*_l - L^*_u)^2 + (a^*_l - a^*_u)^2 + (b^*_l - b^*_u)^2}$$

where $L^*$, $a^*$, and $b^*$ are the CIELab coordinates and the subscripts “l” and “u” refer to the lined and unlined samples.

Pictures of the printed paper before and after reinforcement were taken with a Colorview III digital camera (Olympus Corporation, Tokyo, Japan) and the software Cell^D 3.4 (Olympus Soft Imaging System, Version 3.4, GmbH, Münster, Germany).

RESULTS AND DISCUSSION

Visual Appearance of Lined Samples

Images of the reinforced and non-reinforced four-color printed papers are shown in Fig. 2. The upper image shows each test piece printed in each color, and the lower image shows each color print lined with BC or JP sheets. The images were taken before the samples were subjected to accelerated aging. Clear differences can be seen between the fields lined with BC and fields lined with JP sheets.

For each paper and color, the comparison between the JP-reinforced area (bottom) and the non-reinforced area (top) shows that the JP-lining resulted in a non-uniform appearance with a lighter hue. In contrast, only a slight decrease in color hue was observed.
in the areas lined with BC-lining (middle), in which no mottled appearance was detected. This could be because JP is a very low-density white paper sheet, whereas BC films are translucent and one third of the thickness of JP (Santos et al. 2016a).

The change in the visual appearance of the printed areas after reinforcement with BC or JP sheets did not depend on the paper grade, although the ink color had a clear influence. The changes were more visible on the dark colors, and the black printed samples showed higher uneven areas than the other colors. This is important because most bibliographic information is printed with black ink. This finding concurs with earlier results, indicating that JP reduces the readability of texts (Santos et al. 2016b).

**Change in Print Density of Lined Samples**

Figure 3 shows the print density values for each paper and ink color, measured before (p) and after lining with BC (p-BC) or JP (p-JP).

![Figure 3. Print density for each paper and color print: before (p) and after lining with bacterial cellulose (p-BC) or Japanese paper (p-JP). The standard deviation was less than 0.03 in all paper samples.](image-url)
No data are shown for the optical density in unprinted areas because the change in this parameter was minimal, at less than 0.1 in all paper grades. Similarly, the values of print density measured after the lined printed samples that were subjected to accelerated aging are not shown in Fig. 3 because the treatment did not cause a noticeable change in the values of this parameter.

As can be seen in Fig. 3, the samples reinforced with JP showed a high decrease in print density (more than 40%) compared to the original values, but the samples reinforced with BC had similar or slightly lower values than the unlined samples (maximum change of 12%). Analysis of the results by color showed that the black printed samples had the highest decrease in print density after reinforcing. This was more pronounced for the papers reinforced with JP, in which the density decrease was 0.9, approximately half of the target value. Conversely, the changes in the BC-lined black prints were less noticeable (0.4). In the other color prints, reinforcing with BC produced changes in print density that were imperceptible to the human eye (< 0.05), whereas lining with JP caused a very perceptible change in color intensity, with decreases in print density greater than 0.5.

The effect of paper grade was similar in the four samples examined, although the wood-free uncoated paper (WU) showed a slightly higher decrease.

**Fig. 4.** Color differences ($\Delta E^*$) for samples lined with BC or JP; horizontal lines show $\Delta E^*$ values from which color differences are perceptible at a glance. The variation coefficient of $\Delta E^*$ never exceeds 10%.
Evaluation of Color Differences

The color differences observed in CIELab (ΔE*) were used to evaluate how much the printed color was modified by the reinforcing materials studied. The ΔE* values obtained after lining, for each color print and paper, are shown in Fig. 4. To detect the effect of the paper grade, the ΔE* was also obtained on the unprinted areas for all of the papers. In the printing industry, ΔE* values lower than 2 mean color differences are perceptible to a trained eye. The ΔE* values of 2 to 5 are perceptible to average human observer; nevertheless this values are usually considered an acceptable match in commercial reproduction. The ΔE* values of 6 or higher indicate differences perceptible at a glance.

As can be seen in Fig. 4, no noticeable color change was observed (ΔE* < 3.7) when the unprinted papers were lined with JP. In contrast, BC reinforcement caused a slight color change, with ΔE* values of 4.0 to 6.0. This perceptible difference can be explained by the low L* and b* values of BC compared to the JP values (Santos et al. 2016a). However, in printed areas the opposite trend occurred. The color differences in BC-reinforced samples were always smaller than in the samples lined with JP. The color changes after BC lining were slightly noticeable and in some cases (ΔE* < 6) barely perceptible, whereas the JP lining produced a very marked modification of the printing appearance (ΔE* from 10 to 45).

After BC lining, cyan and magenta printing showed no noticeable differences, whereas black and yellow printed areas indicated ΔE* values higher than 10 units. The color prints had similar behaviour after JP-lining but the black and yellow fields showed more remarkable changes and experienced the greatest modifications, with ΔE* values from 33 to 43, for yellow.

The effect of paper grade was similar for all papers. The super-calendered paper (SC) had the lowest ΔE* for each color print. The wood-free, an uncoated paper with a sizing treatment, had the highest ΔE* after lining with both BC and JP. The effect of lining on the printed areas of the two coated papers was similar, regardless of the finish grade (matte or glossy).

Another important parameter was the stability over time of the color print after restoration treatment. To study the durability of the lined printed samples, they were subjected to accelerated aging and then the color differences were calculated in relation to the non-aged color prints without reinforcement (p).

Figure 5 shows the ΔE* values for unlined (a) and lined samples with BC ([BC]a ) and ([JP]a) for each paper and color print. The ΔE* values obtained in the unprinted areas were also included, as these values are useful for understanding the results obtained according to the ink color and paper grade.

After aging (Fig. 5), the unprinted samples with a high amount of mineral fillers in their furnish composition (GC, MC, and SC) showed ΔE* values of approximately 7.0, whereas the writing paper (WU), manufactured with bleached hardwood pulp and small amounts of filler, had a ΔE* of 4.2 (difficult to perceive). This ΔE* value was within the reported range by other authors for uncoated paper (Van der Reyden et al. 1993; Zappala et al. 1996; Bansa and Ishii 1997; Karlovits and Gregor-Svetec 2011).

When the unprinted papers were reinforced with BC, the effect of aging on unprinted areas was noticeable, with color differences that ranged from 10 to 17, whereas the samples reinforced with JP withstood aging better, showing ΔE* values close to 7. A comparison of the unprinted restored samples before and after aging (Figs. 4 and 5) showed...
that the accelerated aging considerably increased the $\Delta E^*$ values for samples lined with BC, whereas JP-lined samples showed only slight increases in this parameter.

The accelerated aging considerably modified the color of solid print, reaching $\Delta E^*$ values higher than 6.0 in most papers and color prints. These values were higher than those reported by Karlovits and Gregor-Svetec (2011) in their study of the durability of convectional offset print and synthetic paper made from polypropylene and calcium carbonate. The reason for this difference could be that the resulting color differences in cellulose papers were due to the aging of both ink and paper.

The results for the restored and aged printed areas are shown in Fig. 5 ([BC]a and [JP]a), which revealed that the aging of lined printed areas was different from unprinted areas. The color prints lined with JP showed higher $\Delta E^*$ values than BC-restored samples for most of the printed samples. In addition, the $\Delta E^*$ values depended on ink color and paper grade.
With regard to ink color, Fig. 5 shows that the JP-lined yellow prints had the highest color differences (ΔE* > 40) compared with their unlined counterpart, followed by prints in black and magenta (ΔE* > 30). For these three colors, the ΔE* values were 10 to 20 points higher than the values obtained for the BC-lined prints. The reinforcing materials revealed quite similar behaviour only for cyan print, with small differences in ΔE* values between the lined and unlined samples. As mentioned when discussing non-aged color prints, the use of JP as a reinforcing material produced very noticeable color changes. However, comparison of the ΔE* values for the restored sample before and after aging (Figs. 4 and 5) showed that the accelerated aging notably increased the ΔE* values for samples lined with BC, whereas JP-lined samples showed only slight increases in this parameter.

In relation to the paper grade, the super-calendered paper (SC) had lower ΔE* values than the other papers for all color prints, as occurred with non-aged samples. This may be because this paper had a low L* value (Table 1).

The results showed that the reinforcing material clearly affected the color changes, and BC had an advantage over JP because it changed the visual appearance of color print less than JP. This meant that restoration of printed papers with JP produced a more noticeable change than BC in the visual appearance.

**Influence of Reinforcing Material on Print Gloss**

Gloss is another variable used to define the print quality in fine paper. Analysis of the print gloss results confirmed that the ink color used in printing had no notable effect on print gloss. Because of this, it was decided to show the range of this parameter in the four colors for unlined and lined samples before and after aging (Table 2). The resulting print gloss differed widely among the papers tested and the reinforcing materials used.

**Table 2. Print Gloss (%) Range for All Color Prints for Unlined, BC-lined and JP-lined Samples before and after Aging**

<table>
<thead>
<tr>
<th></th>
<th>GC</th>
<th>MC</th>
<th>WU</th>
<th>SC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unlined</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-aged</td>
<td>89 to 100</td>
<td>44 to 57</td>
<td>8 to 9</td>
<td>61 to 72</td>
</tr>
<tr>
<td>Aged</td>
<td>88 to 100</td>
<td>43 to 56</td>
<td>6 to 7</td>
<td>57 to 69</td>
</tr>
<tr>
<td>BC-lined</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-aged</td>
<td>44 to 57</td>
<td>40 to 49</td>
<td>29 to 38</td>
<td>33 to 51</td>
</tr>
<tr>
<td>Aged</td>
<td>48 to 62</td>
<td>42 to 52</td>
<td>34 to 40</td>
<td>36 to 54</td>
</tr>
<tr>
<td>JP-lined</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-aged</td>
<td>5 to 6</td>
<td>5 to 6</td>
<td>4 to 5</td>
<td>4 to 5</td>
</tr>
<tr>
<td>Aged</td>
<td>5 to 6</td>
<td>5 to 6</td>
<td>4 to 5</td>
<td>4 to 5</td>
</tr>
</tbody>
</table>

The print gloss values of the different paper grades varied widely. The unlined samples showed print gloss values of 8% for wood-free uncoated paper (WU) to 100% for glossy coated paper, as expected when using paper with various surface finish grades (Ström et al. 2003; Quintana et al. 2012). After aging, the print gloss of the printed samples showed only minor variation.

However, the traditional reinforcing with JP resulted in a print gloss of 4% to 6% for all paper grades. This means that the final appearance of the document will be matte, regardless of the gloss of the original paper. No notable changes in gloss were observed after aging of the JP-lined samples, which typically remained at low levels.

In contrast, when BC was used as the reinforcing material, the final print gloss depended on the paper grade. For glossy papers (GC and SC), BC produced marked
decreases in print gloss (Table 2). The change in this parameter was more noticeable in the glossy-finished coated paper (GC) than the glossy uncoated paper (SC). For matte-finish-coated paper (MC), the gloss of the lined samples was very similar to the gloss of the corresponding printed samples. For the uncoated matte-finish paper (WU), the resulting print gloss of color prints increased between 25% to 35%. Therefore, when BC was used as the reinforcing material, the resulting print gloss depended on the gloss grade of the paper. Accelerated aging did not noticeably modify this parameter.

CONCLUSIONS

1. Printed areas lined with JP showed a non-uniform appearance in color prints, but this mottled appearance was not observed in the restored printed areas lined with BC.

2. Japanese paper markedly reduced the print density (> 0.5 density points), whereas BC decreased this value only slightly. Although the black ink showed the highest differences, accelerated aging did not notably modify the result obtained for non-aged samples.

3. The color differences in restored printed samples were caused by the material used for reinforcement. For all paper and ink colors, the JP-lined printed areas showed higher $\Delta E^*$ values than the BC-lined printed areas, and the SC-paper showed the lowest $\Delta E^*$ values. However, aging had a greater effect on the BC-lined samples. The influence of ink color on $\Delta E^*$ values was also confirmed.

4. When JP was used as the reinforcement material, the resulting appearance of the document was matte, regardless of the gloss of the original paper. Bacterial cellulose reduced the print gloss in coated and/or glossy papers and moderately increased it in matte-printed paper.

5. In the restoration of printed paper, the use of a BC-sheet showed advantages over JP. Bacterial cellulose produced uniform color areas and fewer changes in color appearance, although the color differences increased with aging treatment.

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sucrofermentans cect 7291 focused on its use in degraded paper restoration.”

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