



## Original article

## Under the spotlight: A new tool (artificial light radiation) to bleach paper documents



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## ABSTRACT

This study offers a new tool to restorers to bleach paper documents: artificial light radiation (ALR). It is a controlled method that is an alternative to other highly aggressive procedures that have been applied to cellulose based media. Its main novelty is its intrinsic lighting characteristics which guarantee uniformity and avoid heating the paper. The results of testing this new tool were compared to those of bleaching with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). Each of the two methods was applied to five different types of widely used paper: Arches and Somerset (brands serving for engraving and digital printing), Ingres (drawing), press-type (magazines and newspapers) and Bible-type (historical documents). Once characterised by physico-mechanical, chemical and optical parameters (grammage, thickness, specular gloss, optical properties, air permeance, tensile properties, determining the pH of both surface and aqueous extracts), the different papers were bleached by the two methods according to pre-established criteria. Finally, to determine the evolution and effects of the two treatments, the papers were subjected to accelerated ageing.

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## Introduction

Bleaching is the process of whitening paper by either applying chemical agents or through exposure to sunlight [1]. Its sole purpose, apart from improving durability, is to enhance a document's aesthetic quality. Bleaching by light radiation is closely linked to the manufacture of paper since its introduction in China in 105 BC as this early procedure included exposure to the sun on wooden boards to enhance the whitening of its fibres [2]. The earliest case of resorting to natural solar light for conservation and restoration of graphic work was carried out by De Mayerne in the 17th century [3]. The Industrial Revolution ultimately introduced new bleaching products comprising chemical reagents considered innocuous at that time by the scientific community and professionals. These products nonetheless turned out to be pose serious risks to the physico-chemical integrity of the media. Keiko Keyes, at the International Conference on the Conservation of Documentary, Archival

and Graphic Arts Materials held 1980 in Cambridge, was the first to propose a return to solar light radiation to restore graphic and written heritage [4]. It was during her residence in the early 1980s at the Department of Cooperstown that she first resorted to artificial fluorescent light [3]. This technique not only facilitated a greater control of the procedure, but could be carried out in controlled indoor conditions. Her long exposure of samples to fluorescent lamp light yielded acceptable results that nonetheless turned out to be detrimental to the media. This problem prompted Roy Perkinson [3] to turn to metal halide light which required less exposure and yielded results four times better than their fluorescent counterparts.

The current study, rooted in these historical antecedents, advances a novel technique to achieve optimal whitening by means artificial light radiation which likewise minimises damage to the documents. To corroborate its suitability, the results of the bleaching carried out with this type of light radiation were compared to tests resorting to hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), a tool considered by certain authors as the least harmful oxidising agent [6].

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The effects of bleaching are directly linked to the chemical characteristics of the medium. In the case of this study the media consisted mainly of cellulose, a linear polymer of plant origin formed by the union of glucose monomers through the covalent  $\beta$ -1,4-glucosidic bond [7]. Cellulose, an organic compound, combines carbon, hydrogen and oxygen atoms in a proportion specified by its chemical formula  $(C_6H_{10}O_5)_n$  where the value  $n$  refers to the degree of polymerisation according to molecule size. The higher the level of polymerisation, the higher the molecule's degree of chemical stability [8]. Cellulose chains are linked chemically by hydrogen bonds which play an exceptional role in the structure of the polymer in that they render it hydrophilic, albeit insoluble in water [9]. These chains give rise to the fibrillar structure of paper whose properties depend on the type of pulp used for their manufacture (cotton, linen, hemp, wood fibres, etc.). Cellulose, however, presents two different phases. The first is crystalline, compact and ordered yielding a highly resistant paper, whereas the second corresponds to an amorphous and irregular phase yielding a paper predisposed to degradation [8]. Many of the paper-based media serving over time are characterised by much more complex contents that include, in addition to cellulose, different proportions of hemicelluloses, lignin, sizing and deposits of different origin. Bleaching in fact would be unnecessary as pure cellulose is a material that is hardly altered with the passage of time. Hemicelluloses correspond to a group of heteropolysaccharides made up of short and branched chains of sugar. Their branched nature, in addition to their lower degree of polymerisation, reduces paper stability converting it into a compound that is easier to solubilise and hydrolyse than cellulose [10]. Furthermore, wood pulp gives rise to lignified papers that easily become brittle and quickly darken due to their greater absorption of UV radiation resulting in photo-oxidation [11].

Bleaching in general is the treatment in restoration which serves to counter the yellowing of paper caused by chromophoric groups (Smith 2012: 226). These groups are formed either spontaneously or by certain catalytic factors such as excessive or inappropriate exposure to oxidising products. They also stem from highly polluted storage environments, UV radiation and metallic impurities, which, in conditions of relative humidity, can accelerate oxidation [9]. The chromophore groups are characterised by high acidity and a high content of carbonyl groups ( $>C=O$ ) which during bleaching oxidise yielding soluble carboxyl groups (COOH) [1]. This process requires activation by energy such as light radiation that interacts with the cellulose chains through absorption and reflection.

Paper, when subjected to light radiation, absorbs certain wavelengths (while simultaneously reflecting others) which leads to its characteristic colour [11]. When the energy is sufficient, the chromophoric groups break down and are transformed into a soluble peroxide or free radicals which, upon elimination, bleach the cellulose medium [5]. As the darker spots on the paper absorb more radiation than the surrounding white areas, the remaining unaltered zones continue to be unaffected [12]. The energy afforded by the light can be complemented by that of certain chemical agents such as calcium hydroxide in an aqueous solution ( $Ca(OH)_2$ ) whose dissociation into hydroxyl ions sets in motion the solubilisation of the chromophoric groups [9].

The bleaching agent of the technique resorting to hydrogen peroxide, in turn, is the anion proceeding from its dissociation in an aqueous solution [6]. This free radical breaks the carbonyl groups and transforms them into soluble carboxyl [13] that can be eliminated by reducing the yellowing of the medium.

The findings advanced in this study therefore stem from the comparison of bleaching carried out through controlled artificial light radiation (ALR) to that of hydrogen peroxide ( $H_2O_2$ ). These two techniques were applied to five different types of papers:

Arches, Ingres, Somerset, press and Bible. The objective was to compare the effects of each procedure on samples of each type of paper as well to observe the evolution of the two types of treatments subsequent to accelerated ageing.

## Research aim

The main objective of this study is to corroborate the validity of a novel lighting system serving to bleach historical documents without jeopardising the physical-chemical and mechanical integrity of their cellulosic medium. The experiment designed for this study resorted to five widely used types of paper which were subjected to two treatments, notably artificial light radiation and hydrogen peroxide, an oxidising agent commonly used in restoration. The comparison of the papers after exposure to each of the techniques evidences the validity of the system of artificial light radiation. To determine the evolution of these processes over time, the papers were put through accelerated ageing. The intention of this study is therefore to define a protocol for artificial light radiation bleaching and adapt the conditions to each specific paper type so as to optimise the results. Its ultimate aim is to highlight the multiple advantages of this lighting system to bleach historical documents insofar as it leads to notable optical and aesthetic improvements without damaging the physical-mechanical properties.

## Materials and methods

The types of papers, reagents and tools serving to carry out the experiment are listed in Table 1.

The method of the experiment can be broken down into a series of phases involving the preparation of the paper samples, their bleaching by means of the two techniques and their accelerated ageing (Fig. 1). Physico-mechanical, chemical and optical characterisation of the papers were carried out subsequent to each phase. Analyses and comparisons of the data then led to the conclusions.

### Preparing the paper samples

The experiment were carried out on five samples of each type of paper (A4 format; 210×297mm). The first action, prior to bleaching, was to subject them to an initial artificial ageing to replicate the conditions of many types of old written document. The sole exception was the Bible-type as it had already suffered from natural ageing. The samples were then bleached by each of the two techniques (ALR and  $H_2O_2$ ). After the bleaching process, each was cut into two halves (A5 format; 148×210mm), subjected to another phase of accelerated ageing, and then studies to determine the results. Each of the tests to characterise the samples followed pre-established national and international standards.

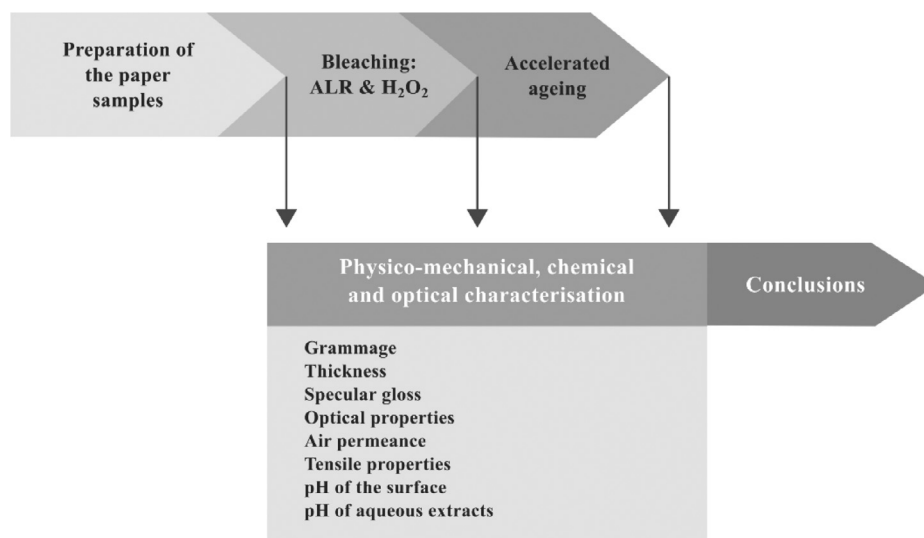
### Bleaching (ALR and $H_2O_2$ )

The controlled exposure to artificial light radiation was carried out while the paper samples were immersed in an aqueous solution of calcium hydroxide. This alkaline solution (pH=9) served to counteract acidification during the process. The pH value was attained by combining 2.9 l of  $H_2O$  and 100 ml of a saturated aqueous solution of  $Ca(OH)_2$  (2,5 g  $Ca(OH)_2$ /l of  $H_2O$ ). The samples were then subjected to ALR for pre-established intervals of either 3, 6 and 9 h. Between the different time trials the solutions of the samples were replaced by a new solution of  $Ca(OH)_2$  to counteract the potential effects of each bleaching on the final results of the experiment.

Following the recommendation of authors such as Schopfer (2012) of placing the light source between 60 and 180 cm, the current test applied the distance of 68 cm above the base of the

**Table 1**  
Materials, reagents and tools serving for the experiment.

<b>Type of paper</b>	Arches	100% cotton fibre Grammage = 280 g/m <sup>2</sup> pH = 7.5–10 Used for printing and digital printing Mechanical pulp with low percentage of cotton
	Ingres	Grammage = 90 g/m <sup>2</sup> Commonly used for drawing 100% cotton fibre
	Somerset	Grammage = 115 g/m <sup>2</sup> pH = 7–9 Used for printing and digital printing 100% TMP (Thermomechanical pulping)
	Press-type (Holmen Plus 68)	Grammage = 48 g/m <sup>2</sup> Used by newspapers and magazines
	Bible-type	Cotton (rag) paper from a naturally aged historical document
<b>Reagents</b>	Calcium hydroxide	Ca(OH) <sub>2</sub> 95% assay PanReac
	Hydrogen peroxide	H <sub>2</sub> O <sub>2</sub> 30% w/v (100 vol.) PanReac
<b>Tools</b>	PRO-LINE Gavita Pro 270 <sup>e</sup> LEP EU (Gravita Professional Lighting)	LEP (Light Emitting Plasma) with an intensity of 2.75–11.1 amps and an input power of 290 W. LUXIM 41.02
	Weight scale Sauter K1200	Meets the requirements of the UNE-EN ISO 536:2021 Standard. Paper and board. To determine grammage
	Digital Micrometre Messmer Büchel (M-172)	Meets the requirements of the UNE-EN ISO 534:2012 Standard. Paper and board. To determine thickness, density and specific volume
	Reflectometer and brightness metre Hunterlab (D48–7)	Meets the requirements of the UNE-EN ISO 8254–1:2009 Standard. Paper and board. To measure specular gloss. Part 1: Brightness of 75° with a convergent beam. Tappi method
	Spectrophotometer Lorentzen-Wettre Elrepho 70	Meets the requirements of the UNE-ISO 57,060:2003 Standard. Pulp, paper and board. To measure the factor of diffuse reflection
	Gurley Instruments Gurley Densometer (4190)	Meets the requirements of the UNE-EN ISO 5636–5:2015 Standard. Paper and board. Measures permeance to air (middle range) Part 5: Gurley method
	Universal Test Systems MTS C43.103	Meets the requirements of the UNE-EN ISO 1924–2:2009 Standard. Paper and board. To determine tensile properties. Part 2: Constant elongation gradient method (20 mm/min)
	pH-metre Crison GLP 22+ Climatic test chamber C-20/350/S (CTS)	With electrodes to measure surfaces and aqueous extracts Working temperature range from –20 °C to 180 °C and relative humidity from 10% to 98%



**Fig. 1.** Schema of the method applied to the experiment.

trays containing the immersed paper. The lamp was turned on a short time ( $\pm 3$  min) before initiating the experiment for it to reach its maximum intensity and level of stability. It was also equipped with a filter to absorb emitting certain particularly harmful wavelengths, notably ultraviolet radiation in spite of the fact that the lighting system itself already restricts these types of emissions. The samples, prior to exposure to the artificial light, were sprayed with

a 50% water-ethanol solution to reduce the surface tension. They were then immersed in the solution under the lamp's light beam for three pre-established intervals (Fig. 2). The treatment was carried out only on the recto (front) of each sample which, after the time had elapsed, were removed and placed between blotting papers.



**Fig. 2.** View of the technique of paper bleaching by means of controlled artificial light radiation (ALR).

The second type of bleaching serving as a contrast with the first was with hydrogen peroxide. As in the previous case, before applying the oxidising agent, the surfaces of the papers were sprayed with a 50% water-ethanol solution to reduce surface tension. The agent itself consisted of the compound mixed with 10% of water to attain a final neutral pH. Since its value was 7.4, no other reagent was added. The  $H_2O_2$  bleach solution was then sprayed on the surface of the samples before leaving them to dry. An identical process was repeated twice to rinse the papers in an aqueous solution of calcium hydroxide with a pH=9. This guaranteed an increase in their basicity after immersion in the solution for 30 min to counteract the slight decrease which took place during the bleaching treatment.

#### Accelerated ageing after bleaching

The paper samples after undergoing both the artificial light radiation and hydrogen peroxide bleaching were subjected to accelerated ageing. This operation consisted of exposing them to extreme environmental conditions to speed up their natural degradation. In this case, the conditions were complete darkness, 65% relative humidity and a temperature of 80 °C over a period of 144 h (6 days) [14].

#### Physico-mechanical, chemical and optical characterisation

In each of the phases of the experiment, a series of tests were carried out to characterise the samples from a physico-mechanical, chemical and optical perspective in order to determine the effects and evolution of each of the bleaching treatments (Fig. 3). These tests were undertaken at the National Institute of Agricultural and Food Research and Technology of Madrid (INIA) following Spanish and International regulations. Once the specimens were conditioned to laboratory values (23 °C and 50% RH), they underwent the following tests: grammage [15], thickness [16], specular gloss [17], optical properties: ISO whiteness [18], coordinates CIELab  $L^*$ ,  $a^*$ ,  $b^*$  and yellowing index [19], opacity [20], air permeance [21], tensile properties [22] and the pH of both the surface and aqueous extracts [22,23].



**Fig. 3.** Photographs of the different actions to characterise the physico-chemical, mechanical and optical features of the paper samples subsequent to the two bleaching (ALR and  $H_2O_2$ ) treatments.

## Discussion

Determining the effectiveness of the bleaching treatments in recovering and maintaining the original characteristics of the paper samples was then undertaken by comparing the bleached and aged samples with examples of untreated, perfectly preserved paper. The most relevant findings of the experiment are presented below in the form of graphs which highlight the variations of the percentages of each of their physico-mechanical, chemical and optical properties compared to those of untreated reference samples. A positive percentage therefore indicates an increase of each variable while a negative percentage designates a decrease among the aged or bleached samples.

#### Arches paper (Fig. 4)

The grammage of the samples of Arches paper suffered a slight decrease during the bleaching and accelerated ageing, whereas their thickness and tensile strength remained unchanged. This implies a certain stability of the medium. Porosity and brightness suffered a general decrease during bleaching regardless of whether the technique was ALR or  $H_2O_2$ . Their values were maintained nonetheless after accelerated ageing. Surface pH measurements reveal hardly any variations throughout any of the phases. The pH of the aqueous extracts, in turn, reveal a downward trend immediately after ALR bleaching which indicate an increase in acidity depending on the time of exposure to the light (from pH=7.5 at 3 h to pH=6.7 at 9 h). The accelerated ageing subsequently decreased the pH of the aqueous extracts. No case attained worrisome pH values that could have affected their preservation thus indicating they remained approximately neutral.

The changes of the physico-mechanical properties of the samples sprayed with hydrogen peroxide are comparable to those of bleaching by ALR for 9 h.

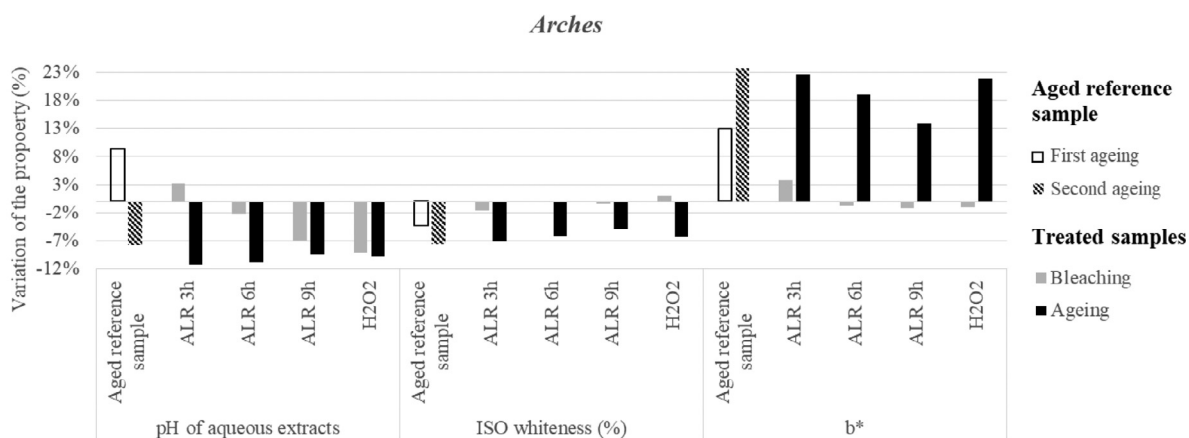


Fig. 4. The most relevant variations of the physical-mechanical, chemical and optical properties of the samples of Arches paper after bleaching and accelerated ageing. Noteworthy is the progressive decrease of pH of the aqueous extracts when increasing the time of exposure to light.

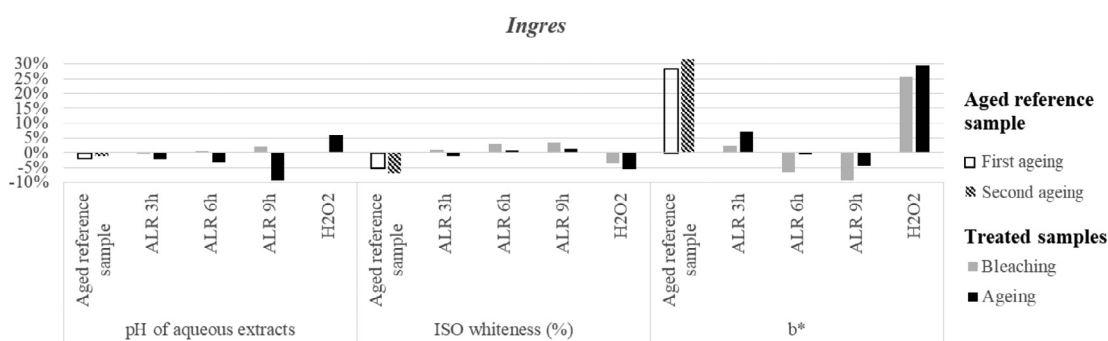


Fig. 5. The most relevant variations of the physical-mechanical, chemical and optical properties of the samples of Ingres paper after bleaching and accelerated ageing. Noteworthy is the low level of effectiveness of the hydrogen peroxide technique as it does not improve whiteness or surface yellowing.

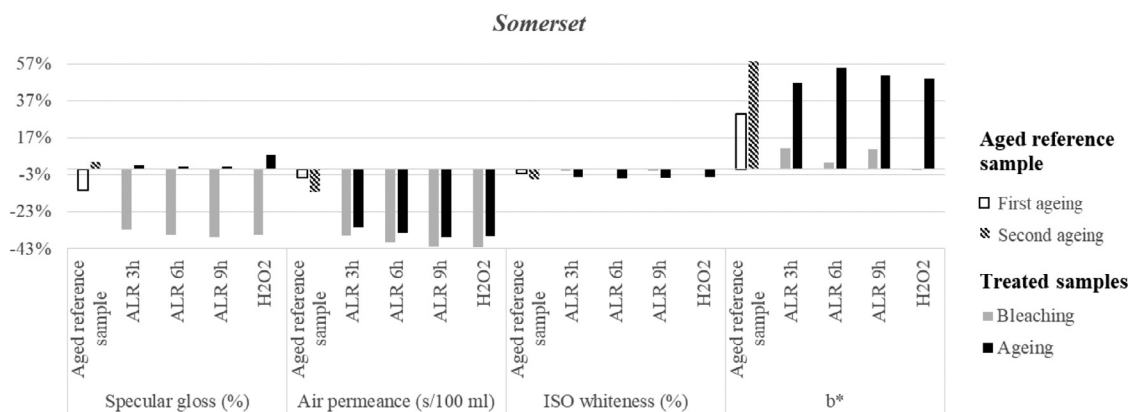


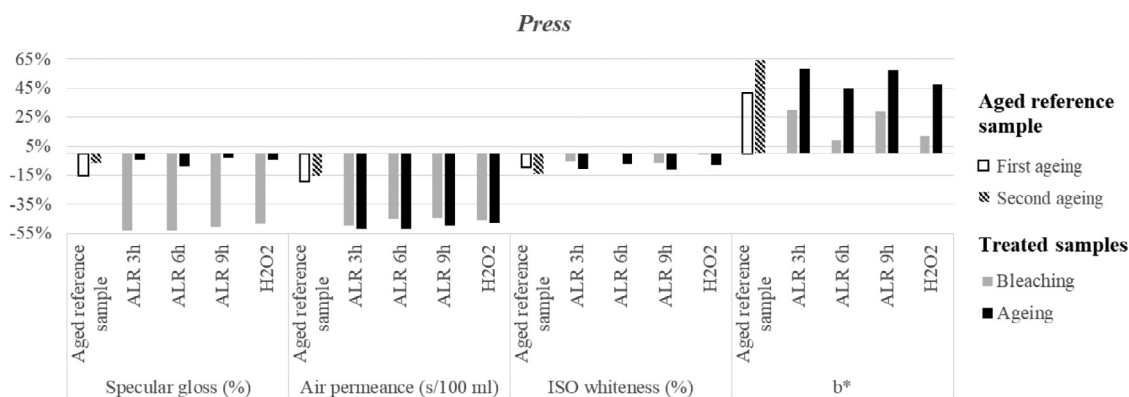
Fig. 6. The most relevant variations of the physical-mechanical, chemical and optical properties of the samples of Somerset paper after bleaching and accelerated ageing. Noteworthy is the progressive decrease of specular gloss and air permeance with the increase of ALR exposure.

The optical properties gleaned from ALR bleaching led to considerable improvements. Whiteness and luminosity, which decreased during ageing, increased again when applying ARL for 6 h. Moreover, surface yellowing ( $b^*$ ) decreased during all the intervals of exposure to light. Although these optical properties are aggravated subsequent to accelerated ageing, they in all cases remain better than those of the reference samples subjected to ageing. This therefore suggests that ALR yields a long-term positive effect. The optimum results of accelerated ageing in terms of the time of ALR bleaching is around 9 h as this interval reveals the least increase in  $b^*$ .

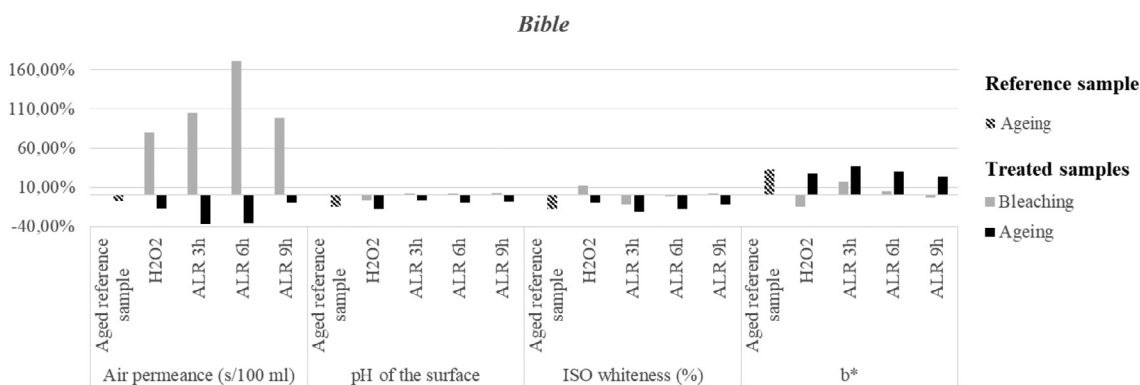
The optical results of the samples sprayed with hydrogen peroxide are equivalent to those of ALR bleaching for 6 h. It is noteworthy nonetheless that the value of the whiteness obtained immediately after the hydrogen peroxide treatment is the highest of all the samples.

*Ingres paper (Fig. 5)*

Grammage and thickness after the ALR bleaching remained, at all times, unchanged. The other properties such as tensile strength, Gurley porosity and brightness were particularly affected by the



**Fig. 7.** The most relevant variations of the physical-mechanical, chemical and optical properties of the samples of Press-type paper after bleaching and accelerated ageing. Noteworthy is the decrease in specular gloss and air permeance associated with the two bleaching treatments.



**Fig. 8.** The most relevant variations of the physical-mechanical, chemical and optical properties of the samples of Bible-type paper after bleaching and accelerated ageing. The results indicate that hydrogen peroxide is more effective treatment for this type of paper.

initial accelerated ageing of the samples and to a lesser extent by the ALR bleaching that, in a way, stabilised the medium. The same applies to the pH of the aqueous extracts, which remained stable after the initial ALR treatment but subsequently decreased after accelerated ageing. This is especially the case of the sample exposed to 9 h of light which attained slightly acidic values (pH=6.5). The decrease of the surface pH of the aged reference sample when compared to the stability of this variable in the treated samples denotes a certain beneficial action of the ALR treatment for the Ingres paper.

The physico-mechanical properties of the Ingres samples bleached with hydrogen peroxide resemble those of exposure to light radiation for 6 and 9 h. It is nonetheless necessary to highlight an increase in grammage occurring both immediately after the H<sub>2</sub>O<sub>2</sub> treatment and after accelerated ageing. This sample likewise suffered the greatest decrease in porosity during ageing.

The results of the measurements of the optical properties are very similar to those of Arches. Whiteness and luminosity increased when applying controlled ALR while surface yellowing decreased (b\*). Accelerated ageing led to a certain deterioration, which was minimal among the samples bleached by ALR. The most effective method from the aesthetic point of view was ALR exposure for 9 h, whereas the least recommended, due to its tendency of colour reversal, was the hydrogen peroxide spray.

#### Somerset paper (Fig. 6)

The grammage of the Somerset paper decreased both after ALR bleaching (up to 5 g/m<sup>2</sup> less than that of the original paper) and during the subsequent accelerated ageing ( $\pm 2$  g/m<sup>2</sup>). Thickness

and tensile strength remained unchanged. Immediately after ALR bleaching, its porosity and brightness decreased progressively as the time of exposure increased. Subsequent to the accelerated ageing there was, nonetheless, a generalised increase in brightness among all samples while the values of porosity remained practically unchanged. The pH values of the surface and aqueous extracts were hardly modified and in no case attained values detrimental to the medium (the level of pH was in no case lower than 7). It is noteworthy that the physico-mechanical properties among the samples sprayed with hydrogen peroxide yielded values similar to those subjected to ALR bleaching between 6 and 9 h.

The ALR treatment yielded positive optical properties with the 3 and 6 h exposure intervals. Whiteness and luminosity decreased after the 9 h timeframe while the yellowing of surfaces increased (b\*). Likewise, this paper suffered a rather marked waning during accelerated ageing, reversing the benefits of the ALR bleaching. There were minimal differences between the aged reference samples and those treated by artificial light indicating that ALR was not effective over the long term. The same occurred with the samples sprayed with hydrogen peroxide as they yielded similar results to those subjected to 6 h of ALR.

#### Press-type paper (Fig. 7)

Bleaching was beneficial to the grammage of press-type paper as it afforded the samples with values close to those of the original paper, especially in the cases of 9 h of ALR exposure and hydrogen peroxide spraying. Each of the two treatments (ALR and H<sub>2</sub>O<sub>2</sub>) yielded unchanged values of thickness and the tensile strength.

Porosity and specular gloss, in turn, decreased notably during each of the bleachings.

The optical properties yielded by ALR bleaching were beneficial at the 3 and 6 h intervals, after which they lost their effectiveness. In addition, the accelerated ageing of the samples treated by both ALR and H<sub>2</sub>O<sub>2</sub> led to a reversion of the colour, that is, a decrease in whiteness and luminosity and an even greater intensification of yellowness (b\*). However, there were certain differences between the aged reference sample and the samples subjected to each of the two treatments that reveal more favourable results subsequent to bleaching. This improved aesthetic among the samples sprayed with hydrogen peroxide is comparable to those subjected to 6 h of ALR exposure.

#### Bible-type paper (Fig. 8)

The grammage of the Bible-type paper after accelerated ageing maintained the improvements achieved immediately after both the ALR and H<sub>2</sub>O<sub>2</sub> bleachings. The thickness remained unchanged while the tensile strength, once again, preserved at all times the changes produced by the treatments. The values of the Gurley porosity test, in turn, decreased after accelerated ageing. With regard to specular gloss, this paper experienced a general decrease after each type of bleaching except for the sample exposed to light for 9 h. The surface pH decreased slightly after the artificial ageing process ( $\pm 0.5$ ) while the pH of the aqueous extracts remained stable throughout the experiment.

Concerning changes to the optical properties, it is noteworthy that the longer the ALR exposure, the better the immediate results. Moreover, those subjected to accelerated ageing revealed better surface whitening and yellowing results as the time of ALR exposure increased. The hydrogen peroxide spray was the most effective bleaching method for this type of medium due to the fact that it yielded optimal whiteness and b\* values throughout all the phases of the experiment.

#### Conclusions

The findings of this study, in general terms, highlight the absence of significant changes to the physico-chemical and mechanical properties of the different papers. They thus reaffirm the validity of artificial light radiation to bleach written documents as this technique avoids damaging the medium. Its effects on the different papers and their properties lead to the following conclusions.

Papers made mainly of cotton fibres such as Arches and Somerset subjected to ALR decrease in grammage presumably due to the dissolution of their sizing components. Their values of thickness and tensile strength remain practically unaltered, whereas their Gurley porosity and brightness undergo compelling transformations. Bleaching each of these papers with ALR produces a gradual decrease in brightness and porosity as exposure to the light increases, changes that remain after accelerated ageing. Otherwise no cases of pH values lower 6.5 were detected among their surface or the aqueous extracts.

ALR bleaching of papers made of mechanical pulp such as Ingres and press-type increases their grammage possibly due to the absorption of compounds. Their properties of thickness and tensile strength in these cases, as in the two previous papers, remain unchanged. The pH values of the press-type, although insignificant, may indicate a certain tendency towards acidification of the medium as a consequence of ALR bleaching.

Considering the absence of a clear trend and the original heterogeneity of the Bible-type paper, it is possible to conclude that it preserves, in general, its initial physico-mechanical properties.

Changes among the optical properties in practically all the ALR bleached samples represent improvements. In general, they reveal

an increase in ISO whiteness (%) and luminosity and a decrease of surface yellowing (b\*). Optimal results immediately after bleaching are obtained after 6 h of ALR exposure, at which point the treatment slows down its effect on the cellulose medium. Hence applying ALR for 9 h is not as effective. However, after accelerated ageing, the specimens exposed to light for 9 h are those that best preserved and in this case the treatment is more durable. It is noteworthy, in turn, that the papers that reveal the most aesthetic deterioration through accelerated ageing are the Ingres and press-types due their contents of lignin and other chemical compounds.

Comparison of the artificial light radiation method with that of hydrogen peroxide reveals the main disadvantage of this second technique as it not only damages certain properties, but has a tendency to revert colour (i.e. towards yellowing) after accelerated ageing. Ingres is the medium most often mechanically and optically affected by the hydrogen peroxide.

Based on the different findings, this study confirms that artificial light radiation is a viable tool for bleaching all five types of paper. In addition, the findings of the search for a balance between aesthetic improvements and preserving the physico-mechanical qualities indicate that the ideal type of ALR is a controlled exposure lasting 3 h. Although this time interval does not yield optimal results, it does offer the greatest aesthetic improvement to the samples and, although its effect is not as long-lasting when compared to that of 9 h, it avoids deterioration of other physico-mechanical properties.

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#### Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:[10.1016/j.culher.2021.10.005](https://doi.org/10.1016/j.culher.2021.10.005).

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