



## Effects of Photo-oxidation on the Properties of Hemp Office Papers

Ivana Plazonić<sup>1\*</sup>, Vesna Džimbeg-Malčić<sup>1</sup>, Irena Bates<sup>1</sup>, Željka Barbarić-Mikočević<sup>1</sup>

<sup>1</sup>Faculty of Graphic Arts, University of Zagreb, Getaldićeva 2, Zagreb, 10 000, Croatia

**Abstract.** This research observes changes in the properties of commercially available hemp office papers exposed to photo-oxidation for artificial aging. In order to accelerate the changes that naturally occur in paper, photo-oxidation was performed using a Xenon light source. The electromagnetic radiation was administered in doses of 550 W/m<sup>2</sup> over 24 hours with a temperature of 60°C. Three types of hemp office papers that differ in chemical composition and manufacturing process were used as samples. The strength properties (tensile index, elongation at break, tear index), surface properties (Bendtsen roughness, Bekk smoothness), pH of paper extracts, and optical properties were observed. The listed properties of artificially aged samples were compared to those of unaged commercial paper samples. The results show that, after only 24 hours, accelerated aging with a Xenon arc lamp affects all observed paper properties. For all analyzed office papers, the values of the tensile index, Bekk smoothness, and pH of paper extracts decreased, while the values of elongation break, tear index, and Bendtsen roughness increased due accelerated aging. Regarding the CIE L\*a\*b\* color space value, lightness (L\*) generally decreased after the aging treatment, and all paper samples became less reddish (a\* decreased) and more yellow (b\* increased). The obtained results of this research reveal that pulp with hemp fibers can produce higher-quality office paper that is more stable against light and temperature influences than those made with post-consumer fibers. However, to increase the durability of manufactured paper, hemp fibers must be bleached.

**Keywords:** Accelerated aging; Hemp; Office paper; Xenon light source

### 1. Introduction

About 92% of global paper production processes depend upon wood as a traditional raw material (Fahmy et al., 2017). However, wood fiber sources are insufficient, and additional fiber is needed in the paper industry (Miao et al., 2014). Many non-wood plants are used as alternative sources only on an experimental basis, although some are used in commercial paper mills. Generally, straw is the largest source of non-wood fiber in the world, but cotton, hemp, sisal, and kenaf are becoming increasingly important sources as well. Despite the wide variety of cellulose fibers from different origins, it is very important that the fiber selected for production provides paper of the required quality. Therefore, many researchers have blended fiber pulp to achieve the desired properties of paper (Tripathi et al., 2013; Nassar et al., 2015).

Due to its low tetrahydrocannabinol content (< 0.2%), *Cannabis sativa* has potential for hemp cellulose pulp and paper production (Danielewicz and Surma-Ślusarska, 2010; Miao et al., 2014). The hemp used for the pulp and paper industries features strong and long

\*Corresponding author's email: [ivana.plazonic@grf.unizg.hr](mailto:ivana.plazonic@grf.unizg.hr), Tel.: +385 (0)1 2371 080  
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fibers with low lignin content. In total, 35% of fibers are classified as long bast fibers, and 65% are short core fibers. Hemp fiber for paper production must have a fiber length of 15–55 mm (Azeez, 2018). The hemp plant has numerous advantages over wood as a raw material. Compared to one hectare of forest, one hectare of hemp can produce four times more paper. Hemp can be reused just after four months of cropping, while trees require 20–80 years (Małachowska et al., 2015). This plant can reach a height of 4–5 meters in only 80–150 days when it is classified as mature for fiber harvesting. Industrial hemp, as an annual plant, achieves a yield of 12–14 tons of dry matter per hectare, from which 10–12 tons can be harvested as fiber mass. In whole hemp stems, the content of cellulose is approximately 47% and lignin is 18% (Danielewicz and Surma-Ślusarska, 2017). Industrial hemp provides a high pulping yield, which is of great importance for pulp and paper mills (Danielewicz and Surma-Ślusarska, 2010). The chemicals involved in making hemp paper are much less poisonous than the chemicals used in making wood pulp paper; hemp paper can be whitened with hydrogen peroxide, while wood paper requires toxic bleaching substances such as chlorine or dioxins (Danielewicz and Surma-Ślusarska, 2010). However, this raw material also has some disadvantages. Due to the macroscopic structure, there are great differences along the plant from top to bottom in terms of morphological structure and chemical composition. Therefore, hemp cannot provide fibers, pulp, and, consequently, paper of a consistent quality.

Regardless of the origin of the cellulose fibers used in the paper industry, from the moment of its production, paper is subject to a natural aging process. The aging process can be defined as a sum of all the irreversible chemical and physical processes that occur in organic materials slowly over time. The stability of paper during the aging process is largely determined by its composition and manufacturing process. Paper components can be classified by their origin, chemical structure, and function as fibers (composed mainly of cellulose but also of lignin, hemicelluloses, and other minor constituents), mineral particles (calcium carbonate, kaolin, talc, etc.), natural sizing agents (e.g., starch or rosin) or synthetic ones (e.g., alkyl ketene dimer and alkenyl succinic anhydride), colorants, and other substances (Area and Cheradame, 2011). Only light that is absorbed by a molecule is effective for producing chemical changes. During the natural process of paper aging, pure cellulose does not absorb visible light (over 400 nm), but strongly absorbs ultraviolet (UV) light (under 200 nm). Residues of hemicellulose and other impurities are always present in the cellulose fibers used for commercial-grade papers. Hemicellulose exhibits similar behavior to cellulose, and lignin strongly absorbs in the UV and visible regions (Zervos, 2010). After light absorption, paper, as a printing substrate, may generate chromospheres (e.g., formaldehyde, furan, methoxy quinone, and stilbene) in acid and undergo alkaline hydrolysis and photo-oxidation degradation, which result in paper discoloration (Cui and Chen, 2011). To evaluate the permanence and durability of paper based on its chemical composition, different methods of accelerated aging have been used by researchers. Most of these methods include elevated temperature and constant humidity (moist or dry, depending on the method). Temperatures are between 60°C and 105°C; higher temperatures cannot be applied for testing since the higher the temperature, the more uncertain becomes the extrapolation to ambient conditions (Eckhart, 2014). However, accelerated aging of paper can just partially reproduce the natural aging mechanisms such as hydrolysis, oxidation, and photodeterioration (Zervos, 2010).

The aim of the present work is to highlight the influence of light and temperature on some basic strength, surface, and optic properties of paper made with hemp fibers and, based on the results of the analysis, provide recommendations for the use of hemp fibers in the paper industry.

## 2. Methods

### 2.1. Materials

In this research, three commercially available uncoated hemp office papers with grammage of 90 g/m<sup>2</sup> were used as samples:




*Type 1* – unbleached handmade sustainable paper made from 100% hemp plant fiber,

*Type 2* – non-chlorine bleached handmade sustainable paper made from 100% hemp plant fiber, and

*Type 3* – paper made from a mixture of 75% post-consumer fiber and 25% hemp fiber.

In addition to cellulose fibers, many other inorganic and organic components are added to the paper network structure in different proportions during paper production. Depending on the type of paper, fillers, adhesives, pigments, and binders are added in larger or smaller quantities, thus causing the office paper available on the market to differ in chemical composition (Neimo, 1999; Özcan and Zelzele, 2017). In the worldwide paper industry, the material used as filler differs depending on availability and economic factors. Important considerations when selecting fillers include low cost, favorable optical properties, and compatibility with the paper production process. To gain better insight into the effect of the hemp fibers on the optical stability of office papers, the inorganic chemical compositions of all three of the analyzed types of paper are summarized in Table 1.

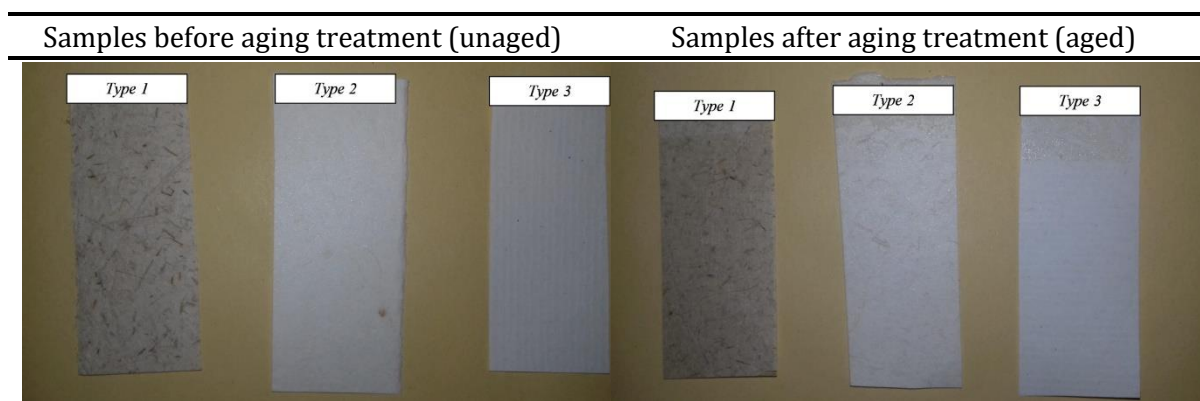
**Table 1** Inorganic chemical composition of hemp office papers (Plazonić et al., 2016)

Paper	Microscopic image at 100× magnification	%			
		Moisture	Ash <sub>900</sub>	CaCO <sub>3</sub>	Clay
<i>Type 1</i>		4.95	1.34	1.14	0.79
<i>Type 2</i>		5.17	1.72	1.49	0.99
<i>Type 3</i>		2.66	4.21	6.35	0.75

### 2.2. Methods

#### 2.2.1. Artificial aging treatment

Samples of hemp office papers were exposed to electromagnetic radiation in a Cofomegra Solarbox 1500e Xenon Test Chamber equipped with a light source (Xenon arc lamp) for 24 hours. The source of electromagnetic radiation emitted a dose of 550 W/m<sup>2</sup> with a temperature of 60°C. One hour of treatment under the Xenon lamp corresponded to one day of natural aging (Debeljak and Gregor-Svetec, 2010). Images of paper samples before and after aging treatment are shown in Table 2.

**Table 2** Images of papers before and after aging treatment

### 2.2.2. Paper analysis

All the properties of hemp office papers were tested before the aging treatment (unaged) and after the aging treatment (aged) in accordance with appropriate standards developed by Technical Association of the Pulp and Paper Industry (TAPPI) and International Organization for Standardization (ISO) (see Table 3).

**Table 3** Properties and methods used for characterization of the samples

Property	Method of analysis
Tensile breaking properties and elongation at break	TAPPI standard method T494 om-06
Tearing resistance	TAPPI standard method T414 om-98
Roughness (air leak method; Bendtsen method)	ISO 8791-2:2013
Smoothness (Bekk method)	ISO 5627:1995
Hydrogen ion concentration of paper extracts (cold extraction method)	TAPPI standard method T 509 om-02
Color (CIE L*a*b* color space, $\Delta E_{00}^*$ )	Standard ASTM (2005) D 2244-93 1993 (ISO/CIE 11664-6, 2014)(E) Colorimetry-Part 6: CIEDE2000 Color-Difference Formula

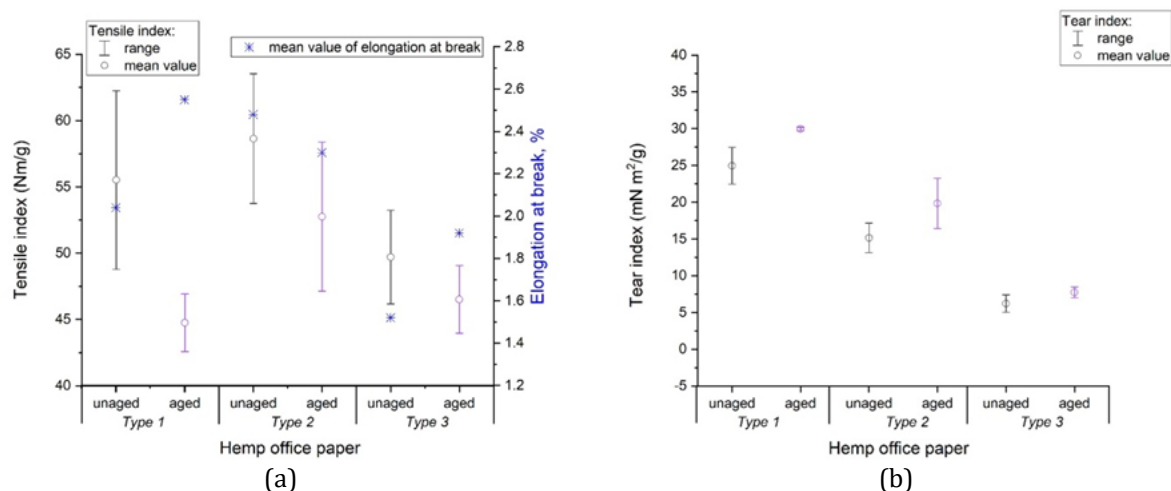
For each sample, a minimum of 10 measurements were made. After summarizing the analysis results, the effect of light on the decay of paper was discussed.

## 3. Results and Discussion

### 3.1. Strength Properties

The strength properties of paper, such as tensile and tear index, can vary significantly depending on the type of fiber used to create the network structure of paper. Not only the type of fiber but also how it was prepared strongly determine the characteristics of paper. These properties are also influenced by chemical changes that cause the paper to lose its stability over time, so its strength properties can serve as indicators of the paper's stability (Caulfield and Gunderson, 1988).

The effect of accelerated aging on the tensile index is illustrated in Figure 1a, which shows that this strength property of paper decreases during accelerated aging. From the tensile index results, it could be concluded that papers with hemp fibers only (Type 1 and Type 2) are stronger than papers in which a small percentage of hemp fibers were added (Type 3).



**Figure 1** Effect of photo-oxidation on the strength properties of papers: (a) tensile index and elongation at break; and (b) tear index

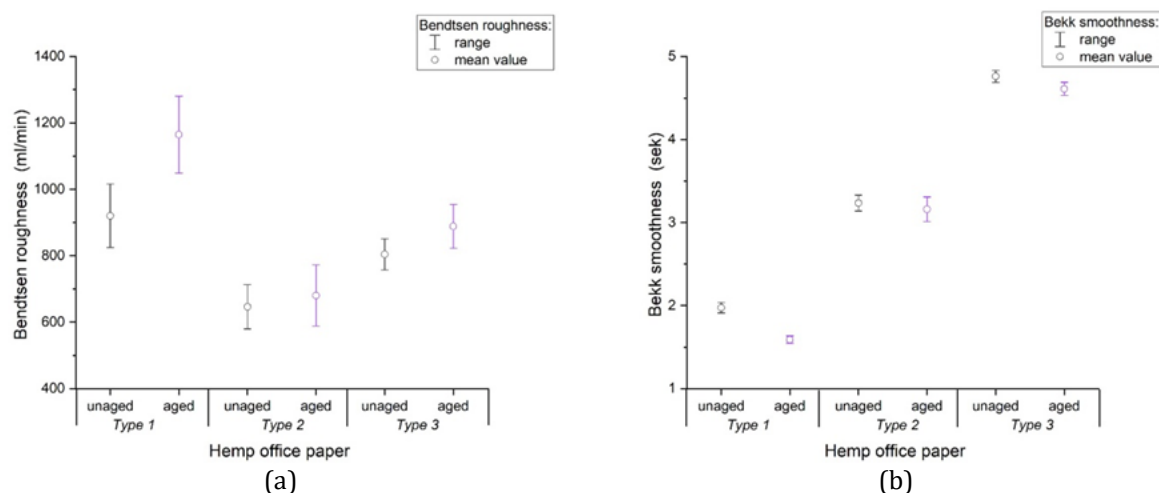
Generally, long fibers provide papers with higher tensile strength than that provided by short fibers (Fagbemi et al. 2014; Koubaa and Koran, 2018). The reasons for strength loss with aging are not completely known, but the property is unquestionably influenced by the intrinsic chemistry, morphology, and structure of individual fibers as well as the network structure of the paper. The highest relative change in the tensile index due accelerated aging was observed for *Type 1*, which was natural unbleached 100% handmade hemp paper (19.4%), while the smallest change was observed for *Type 3*, which contained only 25% hemp fiber (6.4%). The elongations at break were also recorded. Differences in the elongation at break are due to the composition of the paper (Karlovits and Gregor-Svetec, 2012). *Types 1* and *2* contain only hemp fibers that are more expandable and flexible than the post-consumer fibers contained in *Type 3*. Generally, it was found that this property of paper increases with exposure to a Xenon light source.

Unlike paper made with short fibers, papers made from long fibers show much better resistance to tearing (Koubaa and Koran, 2018). Generally, hemp fiber pulp is characterized by high tear resistance (Danielewicz and Surma-Ślusarska, 2017). Hence, papers made only from hemp fibers, either unbleached or bleached (*Type 1* or *Type 2*), have a higher tear index than *Type 3*, which is made with a low proportion of hemp fiber (Figure 1b). Accelerated aging was observed to have a significant effect on the tear index of all analyzed papers. Over 24 hours of aging treatment, the tear index of all papers increased. The highest relative changes in the tear index due accelerated aging were noticed for *Type 3* (41.5%), while the smallest change was observed for *Type 1* (27.8%).

### 3.2. Surface Properties

All analyzed samples are uncoated office papers. As we can see from Figure 2a, the roughness of all papers measured by the Bendtsen air leak method is 646–920 ml/min. It was observed that all papers (*Types 1–3*) became rougher after exposure to the Xenon arc lamp. The highest increase in surface roughness after the accelerated aging process was detected for the *Type 1* sample (up to 26.5%). The other two analyzed papers showed far fewer relative changes in paper surface roughness (*Type 2*: up to 5.3%; *Type 3*: up to 10.5%).

The *Type 1* and *Type 2* samples are handmade papers, and their smoothness is lower than the smoothness of industrially made paper (*Type 3*; Figure 2b). The highest decrease in surface smoothness after the accelerated aging process was detected for the *Type 1* sample (up to 19.7%), while the other two papers showed far fewer relative changes in paper surface smoothness (*Type 2*: up to 2.5%; *Type 3*: up to 3.2%).

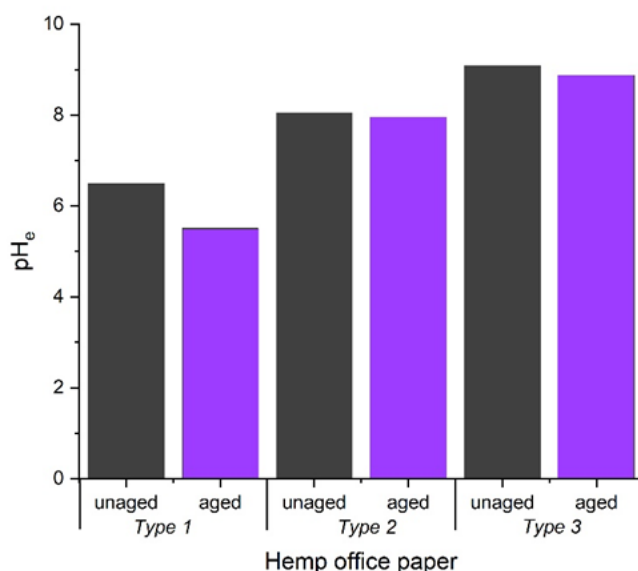


**Figure 2** Effect of photo-oxidation on the surface properties of papers: (a) Bendtsen roughness; and (b) Bekk smoothness

### 3.3. pH

The effect of accelerated aging with a Xenon light source for 24 hours at 60°C on cold extract pH values ( $\text{pH}_e$ ) is illustrated in Figure 3. The stability of paper that is made of organic material as it undergoes the aging process is strongly determined by its composition and manufacturing process. Of all paper properties, acidity/alkalinity is probably the most crucial, as it affects the stability of cellulose, which is the most important structural component (Strlič et al., 2004). Therefore, the pH value of paper is a significant factor determining its stability during aging.

Of all the analyzed papers that contain hemp fibers, *Type 1* is the most acidic ( $\text{pH}_e$  unaged = 6.49), while the remaining two samples, *Type 2* and *Type 3*, are slightly alkaline ( $\text{pH}_e$  unaged *Type 2* = 8.04;  $\text{pH}_e$  unaged *Type 3* = 9.08). The alkalinity of *Type 3* is a consequence of the calcium carbonate ( $\text{CaCO}_3$ ) content (Table 1).  $\text{CaCO}_3$  filler is added during the paper production process in order to achieve alkaline pH and brighter paper in a less expensive way.



**Figure 3** Effect of photo-oxidation on the  $\text{pH}_e$  of papers

Changes in  $pH_e$  (cold extract pH) during photo-oxidation aging of papers were observed. The results in Figure 3 show that after aging treatment, the cold extract pH of all the analyzed papers decreases. The acidity of paper, which is one of the most important intrinsic factors, promotes aging of paper (Zervos, 2010). If lignin is not removed from the pulp (as is the case for *Type 1*), light will encourage acid reactions in paper. The acidity of paper can grow during aging via a combination of oxidation and hydrolysis of cellulose and other constituent parts of paper (Zervos, 2010). Acids that are formed during the aging process in organic materials are formed in alkaline paper as well, but in such papers, formed acids can be neutralized by the alkaline reserve of paper itself. From Figure 3, it is evident that the highest  $pH_e$  reduction due to photo-oxidation aging was achieved in *Type 1* (15.3%), which has the lowest initial pH value of all the papers ( $pH_e = 6.49$ ). For *Type 2* and *Type 3*, the initial pH values of which were slightly alkaline, there was a markedly lower reduction in  $pH_e$  due to accelerated aging ( $\sim 2.5\%$ ).

### 3.4. Optical Properties

To quantify the color changes caused by the aging process, the colorimetric CIE  $L^*a^*b^*$  values of all paper samples before and after accelerated aging were carried out by a X-rite spectrophotometer.  $L^*$  represents lightness (range from 0 for black to 100 for white), while  $a^*$  is the red–green coordinate and  $b^*$  is the yellow–blue coordinate. The latter two are dependent on whether there is a positive or negative value on the corresponding axis. In other words, an increase in the  $a^*$  value means that the color trends toward red, and an increase in the  $b^*$  value means that the color trends toward yellow. Color data were calculated under a standard illuminant D65 (daylight, approximately 6500 K) including 2° standard observers, sphere geometry, specular component, and UV energy. The total difference in color change,  $\Delta E_{00}^*$ , was calculated to assess the results (Table 4).

**Table 4** Effect of photo-oxidation on paper color, defined by the CIE  $L^*a^*b^*$  color space

	Sample	$L^*$	$a^*$	$b^*$	$\Delta E_{00}^*$
<i>Type 1</i>	Unaged	79.51	3.70	18.96	2.01
	Aged	77.48	2.52	19.63	
<i>Type 2</i>	Unaged	94.97	-0.87	9.15	1.34
	Aged	93.15	-0.34	9.51	
<i>Type 3</i>	Unaged	93.61	1.15	1.03	1.88
	Aged	94.34	0.34	2.46	

When exposed to accelerated aging, the surface color of all paper samples changed relative rapidly. After only 24 hours of aging with a Xenon light source,  $L^*$  decreased and  $a^*$  and  $b^*$  increased, except in the *Type 3* sample where a decrease in  $L^*$  values was not observed. All paper samples become less reddish ( $a^*$  decreased) and more yellowish ( $b^*$  increased). Regarding the measured colorimetric CIE  $L^*a^*b^*$  values, the results indicate that under the influence of aging, all the samples became less red and more yellow, especially the *Type 3* sample. Only the *Type 3* sample, which is composed of virgin hemp fibers and post-consumer fibers, had a slightly increased  $L^*$  value. Compared to other office papers (Table 1), the industrial office paper sample (*Type 3*) had the highest content of ash<sub>900°C</sub> (4.21%), and high shares of inorganic filler materials such as  $CaCO_3$  and china clay were added during the paper production process (6.35% of  $CaCO_3$  and 0.75% of china clay). Fillers such as  $CaCO_3$  and china clay are usually embedded in the fiber network to improve the optical properties of paper sheets, including whiteness, brightness, and opacity. The total difference in color change,  $\Delta E_{00}^*$ , do not indicate yellowing, which could be recognized

by a standard observer. As expected, due to its composition (100% natural unbleached hemp fibers), the *Type 1* sample showed the greatest color difference ( $\Delta E_{00}^* = 2.01$ ) after the aging treatment (Plazonić et al., 2018). According to the tolerance definition,  $\Delta E_{00}^* \leq 2$  is classified as a very small noticeable difference for the standard observer, while  $\Delta E_{00}^* \geq 5$  indicates a significant difference that could not be tolerated (Zjakić, 2007). Based on the  $\Delta E_{00}^*$  results, it is evident that office paper formed from bleached hemp pulp (*Type 2*) exhibits better optical stability against artificial aging than papers formed from natural unbleached pulp (*Type 1*). Based on the values of the analyzed optical characteristics presented in Table 4, *Type 2* is the most stable paper.

#### 4. Conclusions

The main conclusions are drawn from the experimental results obtained from exposing hemp office papers to artificial aging by photo-oxidation. The analysis results indicate that hemp fibers can produce high-quality office papers that are more stable against light and temperature influences than those made with post-consumer fibers. However, to increase the durability of manufactured papers, hemp fibers must be bleached. A main purpose of bleaching the pulp is to remove the residual lignin and chromophores inside the pulp, which efficiently absorb UV radiation and cause paper degradation.

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