EFFECTS OF UV, HUMIDITY, AND HIGH TEMPERATURE EXPOSURE ON LINEN FIBERS

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Generally, the most common damaging factors for linen textile materials are the environmental conditions, their handling, and natural decay. Such environmental factors are ultraviolet (UV) radiation, humidity, and high temperature. Therefore, to investigate the effects these factors may cause, an accelerated weathering test was conducted on linen fabrics, using alternating cycles of UV exposure and humidity, along with relatively high temperatures. The effects of this test were investigated using non-destructive and micro-destructive analysis techniques. Scanning Electron Microscopy (SEM) was used to observe any modifications appearing at the surface of the fibers. Energy Dispersive X-Ray Analysis (EDS) was employed in conjunction with SEM for obtaining the spectrum of the chemical elements that were present at the surface of the linen samples. The modifications of functional groups occurring due to the weathering of linen were assessed by Fourier-Transform Infrared Spectroscopy (FT-IR). The color change of the samples was measured with a spectrophotometer. All the acquired information can be used as a starting point for the development of customized environmental parameters for keeping patrimony linen fabrics in museums in optimum conditions, thus preventing further damage. Additionally, the artificially weathered fabrics will be further employed in conservation experiments as substitute for old linen fabrics.

Keywords: linen, weathering, textiles

INTRODUCTION

Textiles have been, for a long time, a significant part of the essential necessities of humankind. The use of textiles commenced with natural fibers of animal and vegetal origin.

There are evidences of the use of linen fibers from more than 30 000 years ago and it is still being valued due to its properties. There are many historically significant linen textiles that are currently found in museums nowadays.

Linen is a bast fiber, meaning that it originates from the inner part of the flax plant stem (*Linum usitatissimum*) (Akin, 2003). The fibers have a diameter of approx. 15 – 17 μm and a clockwise rotation around the longitudinal axis (Markova, 2019). The chemical composition of linen fibers is approx. 71% cellulose, 18.6 – 20.6 hemicellulose, 2.2% lignin, and 1.5% waxes (Friedrich and Breuer, 2015).

MATERIALS AND METHODS

For developing the present work, an undyed linen fabric has been used. The fabric has been divided into six 11x9 cm rectangles. One of the rectangles has been kept as a control sample. The other five fabric rectangles have been submitted to an accelerated weathering process inside an accelerated weathering tester (QUV, Q-Lab). The following weathering method was employed: an 8 h UV exposure cycle at 70°C using fluorescent UVB-313 lamps, followed by a 4 h condensation cycle (60% humidity) at

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50°C. One sample has been collected every three days, the final sample being collected after fifteen days of weathering. The total UV and humidity exposure time for each sample is presented in the following table:

Table 1. UV radiation and humidity exposure time of the linen samples

Sample	UV time (h)	Humidity time (h)
t0 (control)	0	0
t1	48	24
t2	96	48
t3	144	72
t4	192	96
t5	240	120

A Nicolet iS50 (ThermoFisher) Fourier-transform infrared spectroscope (FT-IR) with an attenuated total reflection (ATR) accessory was used to investigate the modifications of the functional groups over a spectral range of 4000-400 cm^{-1} .

A FEI Quanta 200 scanning electron microscope (SEM) has been employed for assessing the morphology of the linen fibers. An Element energy-dispersive X-ray spectroscopy detector (EDS) from EDAX-AMETEK was used in conjunction with the previously mentioned SEM in order to determine the elemental composition of the samples.

The chromatic parameters have been measured with a Datascolor spectrophotometer.

RESULTS AND DISCUSSION

FT-IR

The results for all samples are presented in Figure 1:

Figure 1. Overlapped FT-IR spectra of the linen samples

The structural differences between the samples were minimal. None of the samples presented new IR absorption bands. For all the samples, the IR spectra corresponded with the literature data (Chung, 2004).

The specific band positions for the studied materials in this paper, compared to literature data are presented in Table 2.

Table 2. Absorption band positions in IR for the linen samples, compared to literature

Experimental band position (cm ⁻¹)	Band position reported in literature (cm ⁻¹)	Band attribution
3600-3000	3550-3100	H-bonded OH stretch
2897	2980-2800	C–H stretching
1636	1644	Adsorbed H ₂ O
1427	1429	CH wagging (in-plane bending)
1361	1368	CH bending (deformation stretch)
1334	1337	OH in-plane bending
1314	1316	CH wagging
1280	1281	CH deformation stretch
1247	1247	OH in-plane bending
1203	1203	OH in-plane bending
1159	1160	Asym. Bridge C–O–C
1105	1108	Asym. Bridge C–O–C
1052	1057	Asym. In-plane ring stretch
1028	1030	C–O stretch
897	900	Asym. out-of-phase ring stretch: C ₁ –O–C ₄ ; b glucosidic bond

SEM

The modifications appearing at the surface of the linen fibers with the increasing exposure time to the accelerated weathering conditions can be observed in Figure 2.

Figure 2. SEM micrographs of the control sample (t0) and the artificially aged samples (t1 – t5) at 1000× magnification

With the increasing exposure time to the weathering conditions, the linen fibers have become noticeably damaged. The damage appeared as splits and ruptures, affecting the integrity of the fibers. By the time t5, almost all the fibers were broken. These effects lead to an additional reduction of mechanical properties due to the molecular modifications that resulted from the oxidation at the glycosidic bonds as well as at hydroxyl groups of the cellulose, causing the scission of the macromolecular chains (Kouznetsov *et al.*, 1996).

EDS

This elemental analysis method was mainly employed to quantify the variation of the oxygen/carbon ratio from the main component of the linen fibers – cellulose.

Table 3. Element quantification

Sample	Oxygen (% weight)	Carbon (% weight)	O/C ratio
t0	52.97	47.03	1.13
t1	53.82	46.18	1.17
t2	54.05	45.95	1.18
t3	53.58	46.42	1.15
t4	53.87	46.13	1.17
t5	54.32	45.68	1.19

A slight increase in the O/C ratio can be observed when comparing samples t0 and t5, that can be attributed to oxidative transformations (i.e., aldehyde groups from the linen fibers into carboxyl groups) (Kleinert, 1972).

Chromatic Parameters

The color changes are expressed in a qualitative manner by using three variables: shade, resistance, and luminosity (Colour Terms and Definitions, 2008; Ingamells, 1993). The chromatic parameters indicate the visual differences that may appear when a textile material is aged. The parameter L* represents the brightness of the samples, on a scale from 0 (black) to 100 (white). The DL* parameter indicates the brightness level of the samples. The saturation (C*) represents the intensity of a certain color. The DC* parameter measures the saturation degree of the samples.

Table 4. Chromatic parameters of the linen samples

Sample	L*	C*	DL*	DC*
t0	92.47	1.87	-	-
t1	88.94	1.97	-3.52	0.10
t2	89.24	1.70	-3.22	-0.16
t3	89.05	2.45	-3.42	0.58
t4	88.98	1.39	-3.49	-0.48
t5	89.17	1.52	-3.29	-0.35

All aged samples were darker and less saturated than the control sample. However, sample t3, which was more saturated than the control, is an exception. The visual aspect of the aged linen samples was a color shift towards a redder shade. This effect might be

caused by the photooxidation of the linen fabrics during the ultraviolet exposure (Tera *et al.*, 1985).

CONCLUSIONS

The accelerated weathering experiment that was performed on the linen fabrics revealed, up to a certain point, the effects of ultraviolet radiation and humidity exposure, along with relatively high temperatures. The morphology of the linen fibers was clearly affected by increasing the period of exposure inside the weathering tester, namely, extended fiber ruptures, and splits appeared. These effects have a high negative impact upon the mechanical properties of the linen textiles. At the molecular level though, the modifications imposed by the weathering conditions were not actually significant. However, the visual aspect of the linen samples was affected. The acquired information will be used as a starting point for conservation experiments.

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