



## The Effect of the Accelerated Laboratory Ageing Factors on Linen Textiles Dyed with Turmeric Dye

Mahmoud Abo-Elmaaref <sup>a\*</sup>, Mohammed Maoruf <sup>b</sup>, Wael S. Mohamed <sup>c</sup>,  
Walaa A. Abdel Wahab <sup>d</sup>

<sup>a</sup> conservation department, Faculty of archaeology, Sohag university, Egypt.

<sup>b</sup> Conservation Department, Faculty of Archeology, Sohag University, Egypt.

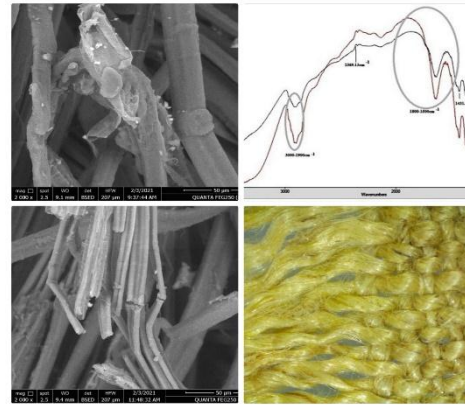
<sup>c</sup> Polymers Department, National Research Centre, Dokki, Giza, Egypt.

<sup>d</sup> Department of Chemistry of Natural and Microbial Products, National Research Centre, Dokki, Giza, Egypt.

### HIGHLIGHTS

- The effect of thermal and light aging processes on the mechanical, physical and chemical properties of dyed linen textiles.
- The effect of light aging processes was greater on the mechanical properties of the dyed fibers.
- The effect of ultraviolet radiation and heat on the morphological and chemical composition of dyed flax fibers

### GRAPHICAL ABSTRACT



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### ABSTRACT

Archaeological linen textiles in uncontrolled museums are exposed to many different damage factors. It is noticed in some Egyptian Museums that the differences in temperature and use of inappropriate light bulbs are the most enemies of dyed historical textiles. This article presents interesting results about the effect thermal and light ageing on optical, chemical and mechanical characterization of dyed linen textiles. New linen textiles are prepared and dyed with Turmeric similar to the ancient linen textiles. The new linen textiles were exposed to artificial heat and light ageing. A close examination was conducted by scanning electron microscope (SEM) and stereo microscope. Fourier transform infrared spectral analysis (FTIR) and CIE-Lab values according to ASTM method D5035 were applied. Among the most prominent results of the study was the

#### Keywords:

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\* Corresponding author: [dr\\_aboelmaaref@yahoo.com](mailto:dr_aboelmaaref@yahoo.com)

appearance of oxidation and hydrolysis bands of cellulosic materials due to ageing process, in addition to the appearance of some bands that express a defect in the chemical structure of cellulosic materials, such as the spectral range at  $435\text{ cm}^{-1}$ .

## 1. Introduction

Archaeological textiles are very sensitive organic materials since they are very prone to damage and deterioration when exposed to uncontrolled museum conditions such as light, heat, humidity, pollution and microorganisms. In other words, they are extremely vulnerable to decay when stored or displayed in inappropriate environmental conditions. So, the conservation intervention is very important in order to improve the properties of the textile objects, enhancing their long-term stability by slowing down the rate of further deterioration. Conservation processes comprise in cleaning, disinfection, consolidation, mounting and storage [1-3]. The process of accelerated aging is a process in which the material is affected by the surrounding environmental factors such as temperatures, light, humidity, biological factors and gases over a period of time from the material's exposure to such conditions [4]. Thermal aging processes are widely used in order to predict the life of polymers in their working conditions based on the decomposition of data collected at high temperatures, and this approach depends on the assumption that the degradation process is governed by one mechanism over the studied temperature range, and provides this type of aging information on the kinetics of slow decomposition reactions over a short period of time [5]. The aging process is carried out by exposing the material in a suitable climatic room to harsh conditions in terms of temperature and humidity for a period of time. During that period, the changes that occur to the material are measured [6]. Experimental decomposition or what is called an accelerated aging is performed on modern materials in an attempt to simulate historical and archaeological materials, and the group of aging processes that take place on textile materials are mostly physiochemical methods including dry thermal aging, wet thermal aging, exposure to ultraviolet rays, decomposition with acids or alkalis, or exposure to air pollutants such as ozone gas  $\text{O}_3$ .

These aging methods is used either in the form of a group together or in a single form and are performed in laboratory conditions [7]. Accelerated aging processes are carried out on materials for achieving three major purposes; the first to establish in a conveniently short time the relative ranking of materials, the second is to estimate potential long-term serviceability of materials systems under exposed condition of use, and the third is to elucidate the chemical reaction involved (the degradation mechanism) and physical consequences [8, 9]. The light aging process is an important tool in the treatment and conservation of archaeological textiles, as it is used to measure and test the stability of treatment and conservation materials, in addition to the effect of exposing dyed textiles in museums to light during museum display [1]. Textile researchers have been investigating the effects of light exposure on cellulosic fabrics for several decades. Most of the earlier results do not provide numerical data useful for the current purpose. Pure cellulose, regardless of the source, should not absorb light of wavelengths greater than 310 nm. Ultraviolet light of shorter wavelengths may lead to chain scission, and wavelengths less than 200 nm can cause further breakdown of the cellulose with the release of small, volatile molecules. In the presence of oxygen, various oxidation products may be obtained [10]. Turmeric is in fact one of the cheapest spices. Although as a dye it is used similarly to saffron, the culinary uses of the two spices should not be confused and should never replace saffron in food dishes. Its use dates back nearly 4000 years, to the Vedic culture in India where it was used as a culinary spice and had some religious significance. The name derives from the Latin (*terra merita*) "meritorious earth" referring to the colour of ground turmeric which resembles a mineral pigment [11]. This research aims to evaluate the effect of aging processes (thermal - light) on the mechanical, physical and chemical properties of turmeric dyed linen textiles.

## 2. Materials and methods

### 2.1. Materials

- Linen textile produced by The Egyptian Textile Industries Company (Dintex) in Cairo.
- Turmeric dye (*Curcuma longa* L) was purchased from the Egyptian local market.
- Potassium aluminum sulphate  $\text{AlK}(\text{SO}_4)_2$  was supplied by Al-Gomhoria Chemicals Company in Cairo and was used as a mordant.

### 2.2. Methods

#### 2.2.1. Dye extraction and dyeing process of linen fibers

The turmeric roots were ground well into a powder form, soaked in water for 24 hours, then heated to boiling point for two hours. The extract was left to cool and then filtered well until a clear and transparent color of dye was obtained. For the dyeing process, a percentage of the solution that was previously filtered from turmeric roots was used (per 1 gram of the dye we use bath volume of 20 ml), then the linen fibers that had previously been boiled well and rinsed to remove any sizing materials and alum  $\text{AlK}(\text{SO}_4)_2$  was added as a mordant. After dyeing, the dye residue was removed by rinsing three times with cold water (5 min, room temperature) [1].

#### 2.2.2. Accelerated thermal and light ageing

Some studies refer that the thermal aging process of 100 °c for 3 days equals 25 years of aging in normal conditions. This is an experimental laboratory attempt for making the characteristics of modern samples like that of the old ones [12, 13, 14] Dyed linen textile was hanged in a temperature-controlled oven “Herous-Germany” on special frames at 140°C for a 72-hours period, which is estimated to be an equivalent to about 200 years of ageing under normal conditions [15].

The light aging process that was performed on the samples, was carried out at the National Institute for Standards (NIS) in Cairo at Metro Loggia Textile Laboratory, using ultraviolet light according to the International

Standard ASTM D6544 - 12 Standard Practice for Preparation of Textiles Prior to Ultraviolet (UV) Transmission Testing. The measurement method was carried out by measuring the radiation levels of the mercury ARC lamp using a radiometer for long ultraviolet rays (NIS 268 UVA), which has the maximum response range at 365 nm, as well as short UVC rays, UVC 268, which has a maximum response at 254 nm, where the distance between the center point on the bulb of the lamp and the detector was 20cm. The average reading for UVA was 5.5616 mW/cm<sup>2</sup> and UVC was 3.0782 mW/cm<sup>2</sup>, and the exposure time of the samples was 5 hours at a relative humidity of 36% ± 4%.

#### 2.2.3. Examinations and analysis

##### Morphological study

The surface morphology of the unaged and aged dyed linen was investigated by SEM and stereo microscopy to show the changes and damage of the fibers. Small samples were taken from different areas of the textiles samples and investigated under Quanta FEG250(NRC) Scanning Electron Microscope SEM [16, 17].

##### Mechanical Measurements

Tensile strength and elongation at break for all the samples under test were measured and evaluated using Shimadzu Universal Tester of type S-500 Japan. The measurements were carried out according to H5KT/130-5000N/[E139-34A.TSX-2.5]-EN ISO 13934-1,1999 Maximum force & Elongation- Strip Method. Recorded temperature was 20°C and relative humidity was 65%. The textiles samples size was 3 x 15 cm [18, 19]. The mechanical properties of dyed linen samples were measured at the National Institute for Standards.

##### Color measurement

Color changes of dyed linen fibers after ageing were examined by measuring the color difference parameter ( $\Delta E$ ). The CIE-Lab values of the dyeing process were measured using a double beam Optimach spectrophotometer (Data color international Spectra flash SF450-UK). The colors are given in CIE Lab coordinates, L\* corresponding coordinate to the brightness (100 = white, 0 = black), while a\* to the red-green coordinate

(positive sign = red, negative sign = green), and  $b^*$  to the yellow–blue coordinate (positive sign = yellow, negative sign = blue). The total color difference  $\Delta E^*$  between two color stimuli  $\Delta E^* = \{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\}^{1/2}$ . [1, 20-22]

### Fourier Transform Infra-Red Spectroscopy with Attenuation Total Reflection (FTIR-ATR)

The structural changes of aged fibers were monitored by Bruker's Vertex 70 – Fourier Transform Infra-Red Spectroscopy with Attenuation Total Reflection (FTIR-ATR) with resolution of  $4 \text{ cm}^{-1}$ . The vibrational bands that appear in the infrared spectra provide information about secondary structure of linen fabrics and information about the chemical functional groups of a sample which leads to a general characterization of the material or even the identification of specific compounds [23, 24].

## 3. Results and Discussion

### 3.1. The effect of ageing conditions on the surface morphology

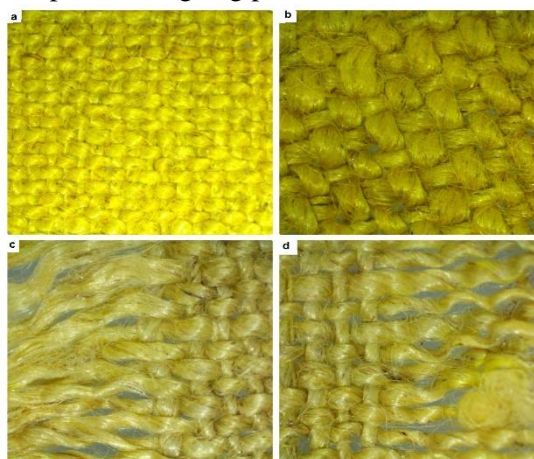
Microscopic examination of the dyed linen samples indicated the darkening of the samples that were exposed to thermal aging, as well as the occurrence of color fading of the samples that were exposed to light ageing by ultraviolet light as shown in Fig. 1.

Scanning electron microscopy (SEM) was used in order to examine the linen fibers before and after the thermal and light aging process. It has been observed that there is a significant change in the morphologic form of linen fiber due to ageing processes. Examination using (SEM) also revealed the occurrence of severe drying and exfoliation of the turmeric-dyed linen samples as a result of thermal aging, as well as the occurrence of breakage in the fibers as a result of exposure to light aging as shown in Fig. 2.

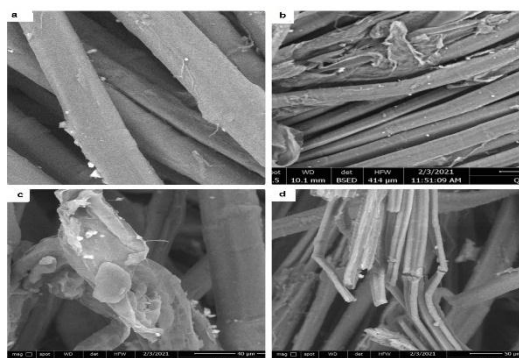
### 3.2. The effect of ageing conditions on the Mechanical Measurements

The literature refers to the changes in the tensile strength as a mechanical property, that not only reflects the changes in crystalline orientation and the chemical structure of the polymer system, but also its morphology. Fig. 3 shows the effect of ageing process on

the tensile strength and elongation of linen fabrics before and after thermal and light ageing. It is noticed that there is a decrease of tensile strength and elongation of linen samples after ageing process.



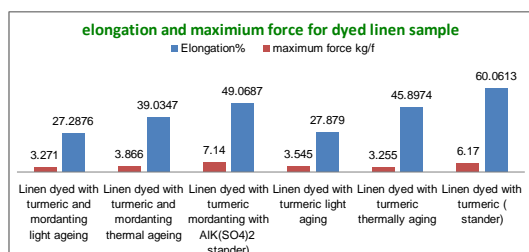
**Fig (1) A stereoscopic image with a magnification of 10x30x illustrating the linen samples dyed with turmeric, where (a) represents the standard sample, (b) represents a thermally aged linen sample with some darkening, (c, d) represent light ageing of linen sample illustrating the fading process of the dye due to exposure to ultraviolet light.**



**Fig (2) SEM image where (a) Dyed linen (b, c) shows the severe damage to the fibers resulting from the thermal aging process, (d) represents the effect of light aging processes on the fibers represented by the occurrence of breakage of the fibers**

The tensile strength and elongation of the dyed linen samples before ageing was

60.0613 kg/f and 6.17%. While, the tensile strength and elongation of the same samples after thermal and light ageing were 45.8974 kg/f, 3.255%, 27.879 kg/f and 3.545% respectively. The tensile strength and elongation of the dyed linen sample after mordanting with alum before ageing was 49.0687kg/f and 7.14%, while the tensile strength and elongation of the same samples after thermal and light ageing was 39.0347 kg/f, 3.866%, 27.2876 kg/f and 3.271% respectively.



**Fig (3) The effect of thermal and light aging processes on the tensile strength and elongation of turmeric dyed linen samples**

### 3.3. The effect of ageing process on the color measurement

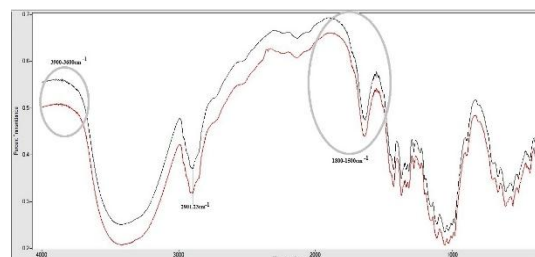
The results (Table 1) show the effect of ageing processes on dyed linen samples. These results can be explained as thermal and light ageing increases oxidation. On oxidation, the side groups of the polymers turn into chromophoric groups (probably quinones) causing the material to develop yellow, brown, grey or light pink color. The results of (Table 1) showed an increase in the total color change values  $\Delta E^*$  of the linen samples dyed with turmeric and ageing with light, where the total color change value ( $\Delta E^*$ ) of the light aged linen sample was 1.52, and this value decreased in the dyed linen sample after mordanting with alum, as the  $\Delta E^*$  value of the sample reached 0.99.

### 3.4. Fourier Transform Infra-Red Spectroscopy

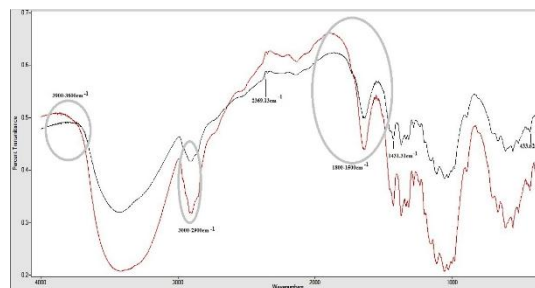
Fourier transform infrared spectral analysis (FTIR), was used to study the chemical structure changes in linen fibers after ageing. The positions of the bands at  $1500\text{ cm}^{-1}$  to  $1900\text{ cm}^{-1}$  indicate the conformations of cellulose degradation [25]. The bands at  $1620\text{ cm}^{-1}$  to  $1730\text{ cm}^{-1}$  represent the carboxylic group, aldehyde group, and conjugated car-

bonyl respectively. While the positions of  $1620\text{ cm}^{-1}$  to  $1710\text{ cm}^{-1}$  indicate to the presence of the ketone group and conjugated carbonyl respectively [26]. The FTIR spectrum of the aged sample showed an increase in vibration of intensity in the spectral range between  $1800\text{--}1500\text{ cm}^{-1}$ , which is a characteristic range for the degradation products of cellulosic materials. Table 2 shows the most important vibrations in this spectral range.

It is also noticed in the thermally aged linen sample that the spectral range  $2901.23\text{ cm}^{-1}$ , which is a characteristic range of expansion and vibration in CH appeared, and the intensity of vibration increased at the spectral range  $3900\text{--}3600\text{ cm}^{-1}$  in a simple way and this is observed by increasing the vibration at the ranges  $3805.8\text{ cm}^{-1}$ ,  $3825.75\text{ cm}^{-1}$ ,  $3833.41\text{ cm}^{-1}$ ,  $3849.03\text{ cm}^{-1}$  and  $3855.68\text{ cm}^{-1}$ . The infrared spectrum of the linen sample dyed with turmeric and aged by UV light showed a decrease in the intensity of the sample's permeability, as well as a decrease in the spectral range  $433.62\text{ cm}^{-1}$  which is a range that expresses a change in the crystal structure of the cellulose polymer and a characteristic range of expansion vibration and bending of CH,  $\text{CH}_2$ , OH and CO for bonds in cellulose polymer [27] as shown in Figs. 4 and 5.



**Fig (4) Change in the infrared spectrum of a turmeric dyed linen sample after thermal ageing**



**Fig (5) Change in the infrared spectrum of a turmeric dyed linen sample after light ageing**

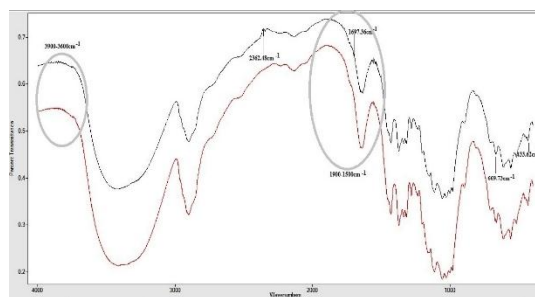
By observing the FTIR spectrum of the linen sample dyed with turmeric and mordanted with alum, it was found that the spectral range of  $433.62\text{cm}^{-1}$  occurred, which is a range in which the decrease reflects the decrease in the rate of cellulose crystallization and the occurrence of a defect in the structure of its crystal structure, as well as an increase in the content of OH-hydroxyl groups [28]. It is also noted that the spectral range  $669.73\text{ cm}^{-1}$  appears as a characteristic domain of sulfate may be due to the use of potassium and aluminum sulfate mordant in the dyeing processes for fibers. Also, by observing the infrared spectrum of the sample the increase in the intensity of vibration at the spectral range  $1900\text{-}1500\text{ cm}^{-1}$  which is a characteristic range for the degradation products of cellulosic materials, is shown in Table 3, the most important vibrations in this spectral range.

The spectral range  $1633\text{ cm}^{-1}$  is a distinctive band that expresses the vibration in the absorbed water inside the cellulose [27], and the spectral range  $1750.52\text{ cm}^{-1}$  is a distinctive range for carbonyl groups. The emergence of the spectral band  $2362.48\text{ cm}^{-1}$ , which is a distinctive band of  $\text{CO}_2$  was adsorbed from the atmosphere as a result of the increase in the sample surface during aging processes. Also, by observing the change in the FTIR spectrum of the linen sample, an increase in the intensity of vibration is observed at the spectral range characteristic of the OH- groups  $3900\text{-}3600\text{ cm}^{-1}$  and the following values show the most important vibrations in this spectral range  $3712.68\text{ cm}^{-1}$ ,  $3755.92\text{ cm}^{-1}$ ,  $3805.8\text{ cm}^{-1}$ ,  $3822.43\text{ cm}^{-1}$ ,  $3849.03\text{ cm}^{-1}$  and  $3859.01\text{ cm}^{-1}$ . Vibration is also observed at the range  $2901.25\text{ cm}^{-1}$  which is a characteristic range of expansion and vibration in CH as shown in Figs. 6 and 7.

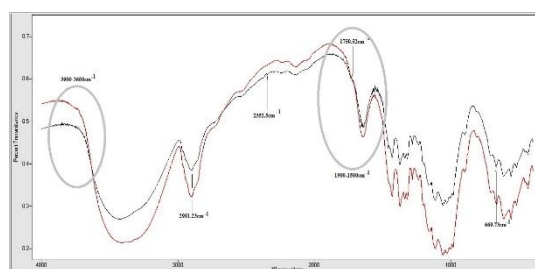
### Conclusion

It is known that archaeological textiles are exposed to damage due to poor display and storage in museums and accelerated aging processes are used to predict such effects in the future. Through the various aging processes (thermal, light) carried out in this study, the mechanical effect was confirmed

represented in the occurrence of a decrease in the tensile and elongation forces of the turmeric-dyed linen samples after being exposed to aging processes. Examination with SEM of the morphological surface of the fibers exposed to aging processes also revealed the occurrence of severe dehydration and exfoliation of the fibers as a result of thermal aging and breakage in the fibers as a result of the samples being exposed to light aging using ultraviolet rays. The results of the analysis with infrared spectroscopy showed an increase in the intensity of vibration in the characteristic range of degradation and oxidation of cellulosic materials  $1900\text{ -}1500\text{ cm}^{-1}$ , in addition to the appearance of bands that express a change in the structure and structure of cellulose such as spectral range  $435\text{ cm}^{-1}$ .



**Fig (6) Change in the infrared spectrum of a turmeric dyed linen sample after mordanting with alum and thermal ageing**



**Fig (7) Change in the infrared spectrum of a turmeric dyed linen sample mordanting with alum with light ageing**

**Table (1) Color difference of linen samples dyed with turmeric**

Color difference of linen samples dyed with turmeric							
Sample	L*	a*	b*	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	$\Delta E^*$
Linen dyed with turmeric	66.39	2.44	34.11	-	-	-	-
Linen dyed / thermally aged	67.29	1.95	34.38	0.90	-0.49	0.27	1.06
Linen dyed /light aged	67.33	2.72	35.27	0.94	0.28	1.16	1.52
Linen dyed with turmeric mordanting with $AlK(SO_4)_2$	68.26	1.52	34.40	-	-	-	-
Linen dyed / mordanting thermally aged	69.21	1.28	33.53	0.95	-0.29	-0.87	1.31
Linen dyed / mordanting light aged	67.76	1.04	33.77	-0.59	-0.48	-0.63	0.99

**Table (2) Vibration in the spectral range of 1500-1800  $cm^{-1}$  characteristic of degradation products of cellulosic materials**

Vibration at spectral range 1800-1500 $cm^{-1}$  for light and thermally aged linen dyed with turmeric

Linen thermally aged		Linen light aged	
1557.88	1634.17	1507.8	1737.25
1567.66	1654.12	1534.4	
1507.8	1720.64	1551.03	
1507.98	1507.8	1561.01	
1514.45		1697.98	

**Table (3) Vibration in the spectral range of 1500-1900  $cm^{-1}$  characteristic of degradation products of cellulosic materials for linen sample after mordanting with alum**

Vibration at spectral range 1900-1500  $cm^{-1}$  for light and thermally aged linen dyed with turmeric and after mordanting with alum

Linen thermally aged		Linen light aged	
1504.47	1713.98	1504.47	1737.26
1541.05	1657.45	1537.73	1770.52
1557.68	1767.19	1557.68	1790.47
1564.33	1787.3	1564.33	
1574.31	1863.64	1567.66	
1640.82	1866.96	1633.49	
1657.45		1654.12	
1664.1		1697.36	

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